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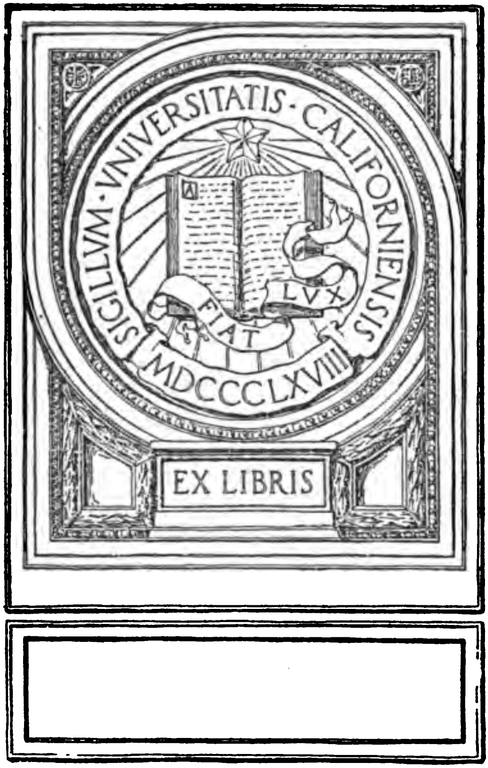
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H. P. TALBOT, PH.D., SC.D., CONSULTING EDITOR

LABORATORY EXERCISES
IN
INORGANIC CHEMISTRY

LABORATORY EXERCISES IN INORGANIC CHEMISTRY

BY

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PREFACE

THIS laboratory manual contains, in addition to the experiments ordinarily found in books of this class, a large number of new experiments that have been designed to illustrate the general principles which are being more and more emphasized in elementary instruction in chemistry. About one-fourth of the book is devoted to the study of gases, liquids, and solids, solutions, thermo- and electro-chemistry, and chemical equilibrium. Directions for a number of inorganic preparations are also given. In designing these experiments the authors have had in mind the student who has had a good training in chemistry in the high school, and who should have his interest stimulated in his college course and should be given the opportunity to extend his experience. Many of the experiments are quantitative; but care has been taken to have the manipulations and apparatus of such a simple character that the exercises can be performed successfully by the average student in a laboratory for beginners.

The experiments to illustrate the properties of metals and their compounds are followed by an outline of a general method for testing a simple salt for the metal and acid radical present. The short time available in the first year for qualitative analysis makes it impossible for the student to master the methods used in the separation of the metallic elements. He can, however, by the simple procedure outlined learn to identify pure salts. The method described has been found to be an excellent introduction to qualitative analysis.

Each experiment is preceded by a "discussion" which emphasizes the principles to be illustrated. The questions incorporated into the directions for the experiments have been selected in such a way that it is necessary for the student to read the discussion in order to answer them. As a result, it is impossible for him to complete the record of his work without understanding what is to be learned from the experiment.

Questions in connection with the laboratory directions serve another purpose. It is the opinion of the authors that the student can not be left to himself in his first experimental work; he must be guided if he is to learn to observe closely and to differentiate the essential from the non-essential. A question in the right place furnishes this guidance and avoids, in most cases, the repetition of the experiment.

The notes of the student consist, in the main, of answers to these numbered questions. The plan makes it possible for the instructor to see at a glance whether the student has understood his work.

The authors are greatly indebted to Raymond E. Neal, Instructor in Chemistry, Simmons College, for valuable suggestions and for the preparation of the drawings, from which the cuts were made.

JAMES F. NORRIS
KENNETH L. MARK

BOSTON, MASS.
April, 1922.

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LABORATORY EXERCISES IN INORGANIC CHEMISTRY

GENERAL DIRECTIONS

The Object of Laboratory Work: Laboratory work is a form of study. A thorough knowledge of elementary chemistry can not be acquired from books alone. Actual first-hand observation of the properties of substances must be made by the student himself, but even these observations will be of small value unless he appreciates the object of the experiment and the principle which it illustrates. Merely "doing" the experiments, that is, mechanically following directions, is as valueless as reading a book without comprehending its meaning. No experiment should be begun until the discussion is thoroughly understood.

Notes: Observations must be recorded immediately. To facilitate clearness and brevity they are to be entered on the blank pages facing the text, as answers to the numbered questions.

The data obtained in quantitative experiments should be carefully labeled and the result of each measurement should be placed on a separate line, as illustrated by the following example in the determination of the percentage of oxygen in potassium chlorate, as carried out in Experiment 11.

Wt. crucible empty	20.30	gms.
Wt. crucible and $KClO_3$	22.03	
Wt. $KClO_3$	1.73	
Wt. crucible and $KClO_3$ after first heating	21.42	

Wt. crucible and $KClO_3$ after second heating......	21.35
Wt. crucible and $KClO_3$ after third heating.......	21.35
Loss of weight = weight of oxygen..............	0.68
Percentage of oxygen in $KClO_3$..................	39.3

The data must be entered at once directly in the book, never on loose pieces of paper. All calculations should be made in the book.

Individual Work: All experiments should be done entirely independently. A student can get no more out of his laboratory exercises than he puts into them. If he allows another student to perform the experiments for him or copies his notes, he is deriving no benefit from his work, he is wasting his time and, moreover, he is dishonest.

Use of the Text Book: The numbers given in the references are those of the paragraphs in "A Text Book of Inorganic Chemistry," by James F. Norris. The text book should be brought to the laboratory at each exercise, the paragraphs pertaining to the exercise of the day should be read, and reference to the book should be made whenever the student is in doubt.

Repetition of Experiments: If the results obtained are not those which you have been led to expect, search for the cause, making sure particularly that you have followed the directions precisely. Not until a possible cause of error has been detected should the experiment be repeated.

Cleanliness: Apparatus must be clean in order to obtain trustworthy results. Always have a clean towel at hand.

The desk top should be frequently wiped with a wet sponge and any material which is spilled upon it should be washed off immediately with plenty of water.

General apparatus must be cleaned and returned to its place at the close of each exercise.

Materials: All the materials required for an experiment and listed in the text should be obtained before beginning work. It is assumed that each desk is supplied with solutions of dilute hydrochloric, sulphuric, and nitric acids, of sodium and ammonium hydroxides, and with concentrated sulphuric acid. These substances therefore are not included in the lists. It is also assumed that each student is provided with the set of apparatus given in the appendix and only additional apparatus is included in the list of materials.

Only such amounts of substances as are actually required should be obtained. Stock bottles should never be carried to the student's desk. Solids may be measured out on a watch glass or on a piece of filter paper and liquids may be carried in a test tube or beaker.

Always read the label on a stock bottle before taking material from it, and verify your reading by rereading after you have taken the substance.

Unused material must never be returned to the stock bottle but thrown away into the crock or sink.

Waste Material: Solids, particularly matches, should be disposed of in the crock, not in the sink. Liquids should be poured into the sink, and in the case of concentrated acids, the sink should be flushed immediately with a large amount of water.

Accidents: Any corrosive liquid on the skin should be washed off *immediately* with plenty of water.

Burns and cuts, even when slight, should be reported to an instructor for treatment.

Acid spilled upon the clothing should be neutralized with ammonia.

Fires should be extinguished by throwing a wet towel over the blaze.

LABORATORY PROCESSES

1. Heating: The Bunsen burner is lighted by first turning on the gas and then applying a burning match. This order of procedure allows time for the air in the tube and burner to be expelled while the match is being "struck." The burner should always be lighted before it is placed under the apparatus to be heated, never after it has been put in position.

The character of the flame is regulated by adjusting the quantity of air which enters through the holes at the bottom of the burner. Ordinarily a clear bluish flame should be used. Sometimes the flame "strikes back," that is, begins to burn at the base where the air enters. The occurrence is usually due to the admission of too much air. Turn off the gas, and after the burner has cooled, diminish the amount of air by adjusting the movable ring and relight the burner.

Thick vessels, like a bottle or a mortar, should never be heated because the uneven expansion of the material causes them to break.

Porcelain evaporating dishes and crucibles can be heated directly in the flame, but beakers should rest on a piece of wire gauze.

Test tubes should be held in the test tube holder. When a liquid is being heated, the test tube should be inclined and held in such a position that the flame strikes the glass opposite the upper part of the liquid. The tube should be slightly shaken constantly. If the tube is held still and the bottom heated, a large amount of steam may be formed suddenly and throw the contents out of the tube.

All vessels must be dry on the outside when heated; otherwise they may crack.

2. **Filtering**: Fold a disc of filter paper just in halves and then again in quarters. Open one of the segments, leaving three thicknesses of paper on one side and one on the other. A paper cone will be formed which should be placed in a glass funnel. If water solutions are to be filtered, wet the paper and press it firmly to the glass with your finger. When a filtration is to be made into a beaker or an evaporating dish, the funnel should be supported by a ring or by a filter arm and the apparatus adjusted so that the stem of the funnel touches the side of the vessel which is to catch the liquid which comes through; otherwise the liquid will spatter out as it falls.

3. **Drying Test Tubes**: Slip the washed test tube over the end of a glass tube about 1 foot in length and, inclining it mouth downward, wave it back and forth through the flame of a burner. When the test tube is hot blow through the glass tube for an instant. Repeat the procedure if necessary.

4. Preparing Glass Tubing: (*a*) *Cutting:* To cut a piece of glass tubing, first make a slight scratch on the tube with the edge of a triangular file. Then, holding the tube in both hands, place the thumbs together against the tube on the side opposite the scratch. Press the tube as though bending it away from the scratch. A clean break should result. The broken edge of the tube will be very sharp and must *always* be smoothed by holding the end of the tube in the flame until it becomes a dull red color. This process, which is called "fire polishing," causes the sharp edges of the glass to melt and thereby become smooth.

(*b*) *Bending:* A good bend is a smooth curve rather than a sharp angle. Several inches of the tube, therefore, must be involved in the bend, and it becomes necessary to soften the glass by heating it in a wide flame. Put a "flame spreader" on the top of the Bunsen burner and close the holes at the bottom of the burner so as to give a luminous flame. Hold the tube in the yellow part of the flame, letting one end rest lightly between the fingers and thumb of the left hand and turning the tube slowly, but constantly, in one direction with the fingers and thumb of the right hand. When the tube feels pliable, remove it from the flame and make a bend of the desired angle.

The following bent tubes should be prepared as they will be used frequently in these experiments: two right-angled bends each leg of which is 10 cm. long; one right-angled bend one leg of which is 10 cm., the other 20 cm. long; one 45° bend one leg of which is 10 cm., the other 15 cm. long; and one 135° bend one leg of which is 10 cm., the other 15 cm. long.

5. Preparing Corks: Before being used corks should be softened. This can be done by means of a press, which is made for this purpose, or the cork can be rolled on the desk while it is being pressed firmly by means of a block of wood. Sharp cork borers should be used to make the holes of such a size that the tubes to pass through fit snugly. In boring corks it is advisable to push the borer with a rotary motion half way through the cork, taking care that the hole is bored through the center of the cork; the borer is then removed and a hole made from the center of the other end of the cork to meet that first made. By proceeding in this way the edges of the holes on the two sides of the cork will be clean cut, and thus make a tight joint with the tube to be passed through the hole; and the latter will run evenly through the axis of the cork.

6. Setting Up Apparatus: Place the front of the ring stand parallel to the edge of the desk and so that the rod is away from you, not toward you. Arrange the clamps so the main weight of the apparatus is over the base of the stand. The movable jaw of the clamp should be on top. Be careful not to screw the clamp so tight as to crush thin apparatus. Keep vertical lines vertical and horizontal lines horizontal.

In putting a glass tube through a rubber stopper, wet the outside of the tube and then push it in with a twisting motion. Give three distinct pushes, pause long enough to count three and then give three more pushes. If you do not pause, you involuntarily keep pushing harder and harder, often with the result that the tube breaks and the jagged end cuts the hand. Be sure that all stoppers fit tightly and that there are no leaks around the holes of stoppers through which glass tubes pass.

7. Weighing: Objects and material to be weighed fall into two general classes, those in which only an approximate weight, to the nearest gram, is required and those in which an accurate weight, to the nearest centigram, is demanded. Cases in which a convenient amount of material for experimentation is to be weighed out belong to the first class; the quantitative determination of the composition of substances belongs to the second class. Platform scales are used in the first instance; beam balances are necessary in the second.

(*a*) The platform scales must always be tested before using them because they are exposed to the fumes of the laboratory, and easily become rusted. Be sure that the rider on the front of the scales is pushed to the extreme left, and then gently tap one of the platforms. The pointer should swing an equal number of divisions on each side of the middle of the pointer scale and should not come to rest until at least two swings on each side have been completed. If the scales are found to be out of adjustment, an assistant should be called to fix them.

In weighing out material for an experiment the empty container should be placed on the left-hand platform and a brass weight which is presumably too heavy to counterpoise it should be placed on the right-hand platform. When this weight has been tried and found too great, it should be removed and the next smaller one substituted. If this is not heavy enough the next smaller one should be added to it. If this second weight is found to be too great, the next smaller should be substituted for it; or if it is found insufficient, the next smaller should be placed on the platform in addition. Thus all the weights in turn should be tried and rejected or kept as the case demands down to and including the 5-gram weight. The rider on the front of the scales should next be pushed to the right until the scales just balance. The weights on the platform should then be counted up, together with the weight indicated by the position to the rider and the number of grams *immediately* recorded in the notebook. The weights should then be returned to their proper holes in the weight box, beginning with the largest weight; and the number of grams again counted as they are removed from the platform. By this second counting errors are often discovered.

The material to be weighed is now placed in the container and the weight of it and the container together should be determined and recorded in the same way that the weight of the container alone has just been found.

Sometimes objects, such as a piece of wire, which do not require a container, can be weighed directly by placing a disc of filter paper on each platform, testing the scales, putting the material on the paper on the left and counterbalancing with weights on the right, as just described.

Never place any substance directly on the platform without a container or paper to protect it.

(*b*) The beam balances are delicate and should be handled with care. When not in use the beam is supported and held at rest by a mechanism that is controlled by a thumb screw in the front of the balance case. Free the beam by turning the screw, raise the door of the case, and set the balance in motion by waving your hand near one of the pans. The disturbance of air should be enough to set the beam swinging. Draw down the door and observe the swing of the pointer. When the balance is perfectly adjusted, the pointer should swing through an equal number of divisions on each side of the middle of the scale. The balance can be used satisfactorily, however, even when not in perfect adjustment, by taking as the "zero" of the scale that divison mark which is midway between the extremes of the swing. For example, if the pointer swings through two divisions on the left of the middle of the scale and through four divisions on the right of the middle of the scale, the "zero" for this particular occasion is the first mark to the right of the middle. In other words, in making a weighing, the balance is in equilibrium if the pointer swings in the same way it did when nothing was on the pans. When the swing of the pointer on one side of the middle exceeds the swing on the other side by more than four divisions, it is best to call an assistant to make a readjustment.

Always test a balance before using it. After testing turn the thumb screw so that the beam is again supported. Now the object to be weighed may be placed on the left-hand pan and a weight, presumably too heavy, on the other pan. Never place on the pans or remove from them objects or weights while the beam of the balance is free to swing. If the beam is not supported the sudden addition or subtraction of weight will make it lurch and may completely change the adjustment. The weights must always be handled with pincers, as the perspiration on your fingers will tarnish them.

After the object and a weight have been placed on the pans, as just directed, slowly turn the thumb screw and free the beam. Observe whether the weight chosen is too great or too small, again support the beam and make the proper substitution or addition of weights in the same way as directed in the use of the platform scales. Continue the systematic trial of the weights down to the 1-centigram weight. If a certain number of centigrams appears to be insufficient and one more to be too much, choose the one which makes the balance nearer to equilibrium as judged by the swing of the pointer. Count the weights as they lie in the pan and *immediately* make a record in your notebook. The number of centigrams must be recorded even when it is zero; otherwise it will appear that you did not weigh the objects to centigrams.

Never attempt to weigh a warm object. The upward current of warm air arising from it will make it appear lighter than it really is. Never place any material directly on the scale pan. Never add anything to a container or take anything out of it while it rests on the pan.

PHYSICAL AND CHEMICAL CHANGES

EXPERIMENT 1. **Classification of Changes.**

Reference: **5–11.**

DISCUSSION: A substance is recognized by its characteristic properties, such as its color, odor, melting temperature, and behavior when brought in contact with certain other substances, as distinguished from its variable properties, such as size and shape. When a substance is so treated that its characteristic properties disappear and a different set of properties appears, the change is summarized by giving to the substance a new name. Such a change is called a chemical change. On the other hand, when a substance is so treated that only its variable properties are affected, the change is termed physical.

MATERIALS: Magnesium ribbon, paper, asbestos, iodine, sugar, copper wire, iron nail.

DIRECTIONS: (*a*) Hold a piece of magnesium ribbon in the flame of your burner by means of a pair of pincers. **(1)** Have the characteristic properties of the substance changed? **(2)** Has a new substance been formed? **(3)** How should this change be classified? Repeat the experiment using a piece of platinum wire instead of the magnesium ribbon. **(4)** **(5)** **(6)** Answer the same questions as in **(1)** **(2)** **(3)**. **(7)** Tabulate the answers to these questions and to similar questions for the following experiments by heading the columns of the table respectively—Substance, Cause of change, Effect on characteristic properties, Classification—and writing in the list of substances—Magnesium, Platinum, Paper, Asbestos, Iodine, Sugar, Copper, Blue solution.

(*b*) Drop a crystal of iodine into a dry test tube, and heat gently the bottom of the tube until a violet vapor arises and a black deposit appears in the upper part of the tube. Observe the crystals of which this black deposit is composed and then heat them by holding the upper part of the test tube in

the flame. (1) Are the crystals iodine? (2) Give reasons for your conclusion. (3) Are the size and shape of the crystals the same as those of the original iodine? (4) What class of properties underwent change in the transformation of the iodine to the black deposit? Record your results in the table referred to above.

(*c*) Put enough sugar into a test tube to make a layer about 0.5 cm. in thickness and hold the tube in the flame of a burner until no further change takes place. When the tube has cooled, break it in your mortar and examine what has been formed. (1) What is its color? (2) Is it sweet? (3) Will it dissolve in water? (4) Is it sugar?

(*d*) Into a test tube one-fourth full of dilute nitric acid slip a piece of copper wire, warm the liquid gently and allow the action to proceed for 5 or 10 minutes. (1) Has the wire diminished in size? (2) What is the color of the substance into which the copper has been transformed by the action of the acid? Dilute the contents of the test tube by adding an equal volume of water and cool the mixture by holding the tube under the tap. Carefully slip a bright iron nail into the liquid while holding the tube inclined so that the nail will not plunge down and break the bottom of the tube. After 5 minutes remove the nail. (3) With what does the nail appear to be coated? (4) How many changes has the copper undergone? (5) Is the reappearance of a substance after a series of treatments a reason for classifying the changes involved as physical?

MIXTURES AND PURE SUBSTANCES—COMPOUNDS AND ELEMENTS

EXPERIMENT 2. **Mixtures and Pure Substances.**

Reference: **15–16.**

DISCUSSION: Some substances are perfectly homogeneous, that is all particles of the substance have identical characteristic properties. In the case of other substances, different particles have different characteristic properties; these are mixtures. Often the particles of a mixture are so minute and so evenly distributed throughout the whole mass that a substance appears to be homogeneous when in reality it is not. In such cases properties other than appearance must be depended upon to distinguish a pure substance from a mixture. Differences in density, in solubility, in action upon heating, and in behavior in many other tests may prove an apparently pure substance to be in reality a mixture.

MATERIALS: Salt, "Old Dutch Cleanser," three numbered unknown substances.

DIRECTIONS: (*a*) Examine carefully a pinch of salt. (**1**) Do all particles appear to have the same characteristic properties? Add a small pinch of salt to half a test tube of water, holding the tube quietly without shaking it. (**2**) Do all particles appear of the same density as judged by their sinking in or floating upon the water? Shake the test tube vigorously. (**3**) Does the test of solubility show the salt to be made up of different or of similar particles? (**4**) Is salt a pure substance or a mixture? (**5**) State the grounds for your conclusion.

(*b*) Examine some "Old Dutch Cleanser." (**1**) Does it appear to be homogeneous? Put a spatulaful of it into a test tube, fill the test tube half full of water and shake it. (**2**) Does it all dissolve? (**3**) Does it all settle to the bottom of the tube? Pour the material into a filter prepared as directed in paragraph 2 of the Laboratory Processes, page 4, and placed

in a clean test tube. When the liquid has run through the paper, shake the filtrate vigorously. (4) What is formed? Rub a few drops of the liquid between your fingers. (5) What does the substance in the water appear to be? Remove the filter paper from the funnel and spread it out flat. Feel of the material collected on it. (6) Does it feel like the substance in the filtrate? Scrape some of the material from the paper with your spatula, put it in a test tube, add some water and shake it. (7) Is the result the same as with the filtrate? (8) Is "Old Dutch Cleanser" a pure substance or a mixture? (9) What is the evidence?

(*c*) Determine by trying various tests whether the materials marked "No. 1," "No. 2," and "No. 3" are mixtures or pure substances. (1) (2) (3) State in each case what you did and what you concluded.

EXPERIMENT 3. **Formation of a Compound.**

Reference: **16–18.**

DISCUSSIONS: The ingredients of some mixtures may be made to enter into chemical action with each other and produce a new substance, the properties of the substances in the mixture disappearing and a single pure substance with different properties taking the place of the mixture. Substances thus formed are called compound substances or compounds. If all of both constituents of the mixture are to be made to disappear and only a pure compound substance is to be formed, the constituents of the mixture must be in a certain definite proportion to each other. Otherwise all of the substance in the mixture which was present in too large an amount will not be used up and a mixture of this remainder and of the compound will result.

MATERIALS: Powdered iron, powdered sulphur, carbon disulphide.

DIRECTIONS: (*a*) Mix one spatulaful each of powdered sulphur and of fine iron filings by grinding them thoroughly in a mortar. Determine whether a mixture or a pure substance has been obtained by dividing half of the contents of the mortar (the other half should be saved for (*b*)) into three portions in three dry test tubes.

Fill one of the test tubes half full of water, shake it, and allow the solid material to settle. (**1**) In what respect, if any, do different particles of the material differ from one another? (**2**) Is the material a mixture or a pure substance?

To the second test tube add about 10 c.c. of dilute hydrochloric acid and warm it until a gas begins to be given off. (Smell the gas in order to compare the odor with that of a gas given off in a later experiment under (*b*)). (**3**) Describe any observation of the action of the acid on the material which will aid you to decide by this means whether the material is a mixture or a pure substance.

To the third test tube add about 5 c.c. of carbon disulphide and shake it. PRECAUTION: *Carbon disulphide is very inflammable. It must never be brought near a flame. All experiments involving its use must be done under a hood where there is no burner. Any carbon disulphide left in a test tube or other apparatus after performing an experiment must not be thrown into a sink or crock but emptied into a bottle provided for the purpose and kept in a hood.* Filter the contents of the test tube, collect the filtrate in an evaporating dish, and allow the carbon disulphide to evaporate spontaneously. Examine what is left in the dish. (**4**) What is the substance? (**5**) What is left on the filter paper? (**6**) Does the action of carbon disulphide on the material show it to be a mixture or a pure substance? (**7**) How does it show this?

(*b*) Place the remaining half of the material in the mortar in a dry test tube and hold the lower end of the tube in the flame until it begins to glow. At once remove the tube from the flame and observe the progress of the action through the material. (**1**) What is the evidence that action is taking place? (**2**) What kind of a change does this phenomenon indicate? When the action has ceased, again heat the lower end of the tube strongly for 2 minutes. This is done to drive into the upper part of the tube any sulphur which may not have acted. When the tube has cooled, wrap it in cloth and break it by a sharp blow with a pestle. Grind the black mass which was formed in the bottom of the tube in a mortar and place a third of it in each of three dry test tubes. Perform with this material

three experiments similar to those which you did in (*a*) with water, with hydrochloric acid, and with carbon disulphide. (**3**) What does the action of the water and of the acid indicate as to whether the material is a pure substance or a mixture? (**4**) How does it show this? (**5**) Does the odor of the gas given off upon treatment with acid show that the material is a new substance? (**6**) How? (**7**) What does the action of carbon disulphide prove about the material? (**8**) What is your final conclusion as to the purity of the material? (**9**) As to the class of substances to which it belongs? (**10**) What kind of a change is involved when a mixture is changed to a compound? (**11**) What evidence was there in this experiment of such a change taking place?

EXPERIMENT 4. **Decomposition of a Compound.**

Reference: **19–20.**

DISCUSSION: The reverse of the process illustrated in Experiment 3 can be brought about, that is, certain pure substances may undergo chemical changes by which a mixture of two or more substances are produced. Some pure substances, however, can not be decomposed. These are called elementary substances.

MATERIALS: Oxide of mercury, a small splinter (a match stick is not satisfactory, as the wood has usually been treated chemically).

DIRECTIONS: Fill an ignition tube about one-quarter full of oxide of mercury. Grasping the tube with the tongs in the left hand, hold the lower end of the tube in the flame, keeping the tube inclined. When a deposit has begun to appear in the upper part of the tube ignite a splinter, held in the right hand, by thrusting it into the flame, extinguish the blaze by blowing on it, and immediately thrust the glowing end into the mouth of the tube. A marked action should be produced upon the glowing splinter. If nothing happens, repeat the test with the splinter, after the oxide of mercury has been heated for a couple of minutes more. (**1**) What happened to the glowing splinter? (**2**) What is the evidence that some gas other than air was in the tube while it was being heated? (The name of this gas is oxygen.) (**3**) What familiar substance do you find deposited

in the upper part of the tube? (4) Did you heat the tube long enough to decompose all the oxide of mercury? (5) Of what substances have you found oxide of mercury to be composed? (6) Name one characteristic property in which oxide of mercury differs from mercury and one in which it differs from oxygen. (7) Are the properties of oxide of mercury the sum of the properties of mercury and oxygen? (8) Is oxide of mercury a pure substance or a mixture? (9) Is it a compound substance or an elementary substance? (10) How does the experiment verify your answer to (9)?

OXYGEN

EXPERIMENT 5. **Preparation of Oxygen.**

Reference: **23–29.**

DISCUSSION: Oxygen can most conveniently be prepared in small amounts, suitable for studying its properties, by the decomposition of some of its compounds. The temperature necessary to produce the decomposition and the expense of the materials are of prime importance in making a choice. Oxide of mercury, for example, which was shown in Experiment 4 to be easily decomposed, so far as temperature is concerned, is too expensive for general use. Sodium nitrate, on the other hand, which is inexpensive, requires a very high temperature for decomposition. Potassium chlorate, however, can be decomposed at a lower temperature than sodium nitrate and is at the same time much less expensive than oxide of mercury. Potassium chlorate, therefore, is usually selected for the preparation of oxygen in the laboratory. Moreover the temperature at which oxygen can be evolved rapidly from potassium chlorate is considerably lowered by the addition of manganese dioxide, which acts as a "catalytic agent."

MATERIALS: Barium dioxide, sodium peroxide, potassium chlorate, manganese dioxide, splinters.

DIRECTIONS: (*a*) *Sources.* (**1**) In what experiment did you obtain oxygen from the oxide of mercury? (**2**) By what means?

Heat some barium dioxide in an ignition tube and test with a glowing splinter, as you did in the experiment with oxide of mercury. The residue left in the tube is barium oxide. (**3**) What is given off upon heating? (**4**) How did you recognize it?

Place in a small, dry test tube enough sodium peroxide to cover the bottom of the tube completely and add about 5 c.c.

of water. Test for oxygen at the mouth of the tube. (5) What test did you use? (6) What was the outcome of the test?

Put enough potassium chlorate in a 6-inch test tube to make a layer about 1 cm. deep. Heat this in the flame and test with a glowing splinter. It is useless to attempt a test until you can see that gas is being formed. (1) What change in appearance occurs when the potassium chlorate is first heated? (2) How can you tell when a gas begins to be formed? (3) What happened finally to the glowing splinter? (4) What does this action prove as to the identity of the gas evolved?

(*b*) *Catalysis.* Into another test tube put as much potassium chlorate as you have just used, and have ready at hand on a spatula some manganese dioxide. Heat the potassium chlorate slowly and *as soon as it has melted,* hold a glowing splinter at the mouth of the tube. Remove the tube from the flame, *immediately* add the manganese dioxide and test again with a glowing splinter. (1) Was the temperature of the pure melted potassium chlorate high enough to decompose it? (2) How do you know? (3) What was given off upon adding the manganese dioxide? (4) What effect does the presence of manganese dioxide have upon the decomposition temperature of potassium chlorate? Careful experiments have proved that no manganese dioxide is used up in this experiment. (5) What technical name is used for a substance which acts as the manganese dioxide does in this case?

(*c*) *Preparation in Quantity.* Set up an apparatus as shown in Fig. 1, page 28, and so placed that the outlet of the trough will be over the sink.

A is an 8-inch test tube provided with a one-hole rubber stopper through which passes a piece of glass tubing *B* which in turn is attached to the longer piece of glass tubing *D* by means of the rubber connector *C*. *E* is a pneumatic trough upon the shelf of which rests a 250 c.c. bottle *F*.

Weigh out, as directed in the general directions on page 6, on pieces of filter paper, 10 grams of potassium chlorate and 5 grams of manganese dioxide. (1) Why is each of these particular substances used? Mix them thoroughly in a beaker

and then pour them into the tube *A*, which should be removed from the ring stand for the purpose. Holding the tube in a horizontal position, shake and roll it until the contents lie evenly distributed along two thirds of its length, and again place it in position. Be careful not to crush the tube by screwing the clamp too tight, and be sure that the stopper, tubes, and connectors all fit so that there are no leaks.

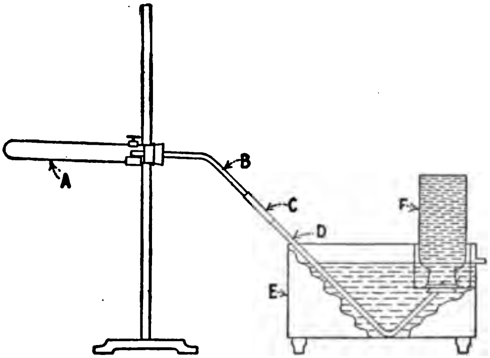

Fig. 1.

Pour enough water into the trough to cover the shelf, over the hole in which is to be placed an inverted bottle full of water. Fill a 250 c.c. bottle brimful of water at the tap and slide a glass plate over the mouth of the bottle so that no bubble of air is included. Invert the bottle in the water of the trough, remove the glass plate, and lift the bottle to its position on the shelf of the trough. Fill three other bottles in a similar way, but leave them on the bottom of the trough ready to be substituted for the one on the shelf, when that has been filled with gas. Adjust the end of the delivery tube under the hole in the shelf. Heat the tube gently by holding the burner in your hand and moving the flame back and forth under that part of the tube which contains the mixture. (The burner should not be held directly under the tube, but to one side at an angle, so that if the tube should break it would not fall upon your hand and burn it.) Bubbles of gas should immediately begin to issue from the delivery tube. (**2**) What is this gas that appears even at the

first warming of the tube? (3) Why does it issue from the tube? (4) If no gas appears, what is wrong with your apparatus? Continue heating the tube until gas is evolved and rises into the bottle at the rate of one or two bubbles per second. When the evolution becomes more rapid than this, remove the flame until the gas comes over more slowly again.

When the first bottle is full of gas, slip a glass plate under the mouth of the bottle while it is still under water and then remove it, keeping it covered with a glass plate or a wet filter paper. Substitute for it on the shelf one of the bottles from the bottom of the trough. In the same way fill all four bottles with gas.

Whenever you stop heating the tube so long that gas ceases to be evolved, disconnect the delivery tube at C. If this is not done water will rise in the tube into the test tube, thus ruining the mixture and often cracking the tube. (5) Why will the water run back into the test tube? (6) What is the gas you have collected? (7) From the decomposition of what compound did it come? (8) Why was the manganese dioxide added?

EXPERIMENT 6. **Properties of Oxygen.**

Reference: **31–33.**

DISCUSSION: Of the many properties which are associated with each substance, only those, as a rule, are studied which are important in showing its characteristic chemical or physical behavior. For example in the case of oxygen, the fact that it will completely fill any space in which it is confined is not emphasized, since this fact is true of all gases and is implied in the statement that oxygen is a gas. On the other hand the action of oxygen in the presence of heated sulphur is different from that of other gases and is therefore chosen as worth studying.

Some particular and striking property, the presence or absence of which may be easily proved, is selected as a "test" or means of identification of the substance among all other substances commonly met.

MATERIALS: Splinter, sulphur, red phosphorus, steel wool, deflagrating spoon.

DIRECTIONS: Referring to the evidence from the last experiment, (1) state whether or not oxygen is freely soluble in water, and (2) the reason for your conclusion.

Thrust a splinter of wood into one of the bottles of oxygen. (3) Does any action take place?

Thrust a glowing (not blazing) splinter into the bottle. Remove the splinter and repeat the action several times. (4) What occurred? (5) What condition is necessary for this action to take place? (6) Did the oxygen burn?

Heat in the flame of the burner a small piece of sulphur in a deflagrating spoon, and when it has begun to burn with a small blue flame, lower it into a bottle of oxygen. (7) What effect has oxygen on burning sulphur? (8) What is formed?

Repeat substituting a little red phosphorus for the sulphur. (9) What is formed?

With the tongs hold a piece of steel wool in the flame and when it is glowing quickly thrust it into a bottle of oxygen. (10) What is formed? (11) What properties of oxygen do the foregoing experiments illustrate? (12) What is a test for oxygen?

EXPERIMENT 7. **Kindling Temperature.**

Reference: **33.**

DISCUSSION: It was evident from Experiment 6 that the substances which were burned in oxygen had to be heated before the combustion started, and that a higher temperature was necessary in the case of some substances, like the steel wool, than in the case of others, like the red phosphorus. All chemical changes take place more rapidly as the temperature is raised. In the case of reactions which evolve heat, either the rate of action may be so slow that the heat is lost to the surroundings as fast as it is produced and thus causes no rise in temperature and speeding up of the action, or the rate may be so rapid that more heat is evolved than is lost, which will cause a rise in temperature and consequent increase in rate of action and again the production of still more heat. When

a combustible substance has been heated from an outside source, as for example by a burner, until the rate of action is so great that the heat evolved from the action is sufficient to maintain the temperature after the burner is removed, then the kindling temperature has been attained. The substance has been kindled and the burning will furnish enough of its own heat to continue the action. Whether or not a given substance always has the same kindling temperature is determined in this experiment.

MATERIALS: Splinter of wood, steel wool, potassium chlorate.

DIRECTIONS: (*a*) Hold the end of a splinter in the flame of a burner until it blazes. Withdraw the splinter and hold the burning end downwards, then upwards. Finally wave the splinter through the air rapidly till it stops burning. (1) What is the technical term used to denote the temperature to which the splinter was heated when it began to burn? (2) Explain in a statement employing this term why the splinter burned more rapidly in one position than in the other, (3) and why the flame was extinguished when the splinter was waved through the air.

(*b*) Hold in the flame, first a mass of steel wool with the tongs and then the end of a steel file. (1) Which one became heated to its kindling temperature? (2) Has steel a definite kindling temperature? (3) Upon what physical condition does the kindling temperature of a substance depend?

(*c*) Heat a little potassium chlorate in a small test tube till you can get a distinct test for oxygen, and then withdraw the tube from the flame. (1) Does oxygen continue to be given off? (2) Does the evolution of oxygen produce heat or does the action appear to require the constant supply of heat? (3) How does the cessation of the action here differ in cause from the cessation of the burning of the splinter in (*a*)? (4) Does the decomposition of potassium chlorate have a kindling temperature? (5) What kind of reactions have a kindling temperature? (6) What kind do not?

HYDROGEN

EXPERIMENT 8. **Preparation of Hydrogen by Various Means.**

Reference: **41–48.**

DISCUSSION: Hydrogen is prepared from its compounds, but the result is not brought about as in the case of oxygen by the decomposition of the compounds by heat. The hydrogen compound is treated with a metal which will unite with the other element or elements of the compound, and thus set free the hydrogen. The readiness with which these actions take place depends upon the particular compounds and metals chosen and upon the temperature and the presence or absence of catalytic agents. Water, acids, and bases are compounds of hydrogen available for use; and many different metals, but not all, may be employed. Certain metals which will not themselves serve to liberate hydrogen from one of its compounds will act as catalytic agents when they are in contact with a metal which is being used for this purpose.

MATERIALS: Sodium, strips of zinc, iron nail, iron filings, aluminium, dilute acetic acid, copper sulphate solution.

DIRECTIONS: (*a*) Fill a small test tube with water and invert it in a pneumatic trough nearly full of water. Obtain from an instructor a small piece of sodium in a *dry* evaporating dish. CAUTION: *Never handle sodium with your fingers, as enough moisture is present on them to cause action. Always use pincers.* With the left hand raise the inverted test tube till its mouth is about an inch below the surface of the water in the trough. With the right hand quickly insert under the mouth of the test tube the piece of sodium held in the pincers. When the action of the sodium and the water has ceased, place your thumb over the mouth of the tube, carry the tube to the burner and uncover it in an inclined position with the mouth downward and close to the flame. (1) What occurred? (2) What

was the gas collected? (3) What was the source, the water or the sodium? (4) How can you recognize hydrogen?

(*b*) Slip a strip of zinc into a test tube half full of water. (1) Is hydrogen evolved? Repeat the experiment using an iron nail instead of zinc. (2) Is hydrogen evolved? (3) What results would have been obtained at much higher temperatures?

(*c*) Set up an apparatus as shown in Fig. 2. *A* is an 8-inch test tube provided with a two-hole rubber stopper, *B* a thistle tube, *C* and *D* bent glass tubes, *E* a rubber connector, *G* a 250 c.c. bottle filled with water, *H* a pneumatic trough.

Slip into the test tube two strips of zinc, replace the stopper and pour down the thistle tube enough dilute hydrochloric acid to fill the test tube a quarter full. When the bottle has become a third full of gas, substitute in its place a small inverted test tube filled with water. When the test tube has become filled with gas, carry it to the flame, with your thumb over its mouth, and test for hydrogen. (1) What was the gas first collected in the bottle? (2) Where did it come from? (3) What was the gas in the test tube? (4) Was it produced from the hydrochloric acid or from the zinc? (5) Of what element is hydrochloric acid a compound?

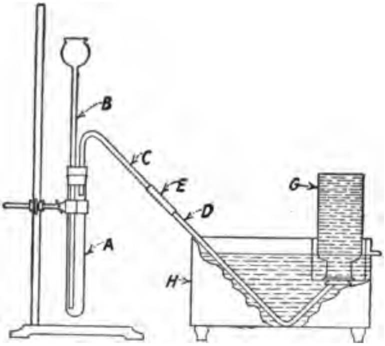

FIG. 2.

Empty the large test tube and repeat the experiment, first substituting dilute sulphuric acid for the hydrochloric acid and then again perform the experiment using acetic acid and

finally using dilute nitric acid. (6) In which cases did you find hydrogen to be evolved? (7) Which acid or acids are best adapted for preparing hydrogen under these conditions?

(*d*) Using the same apparatus as in (*c*), perform experiments which will enable you to answer the following questions. (1) Is hydrogen evolved by the action of hydrochloric acid on iron (use iron filings)? (2) on aluminium? (3) on copper? (4) Which metal or metals are best adapted for preparing hydrogen?

(*e*) Into each of two test tubes standing in the test tube rack put two strips of zinc of the same size and just cover them with dilute sulphuric acid. To one add five drops of copper sulphate solution. The black deposit which appears on the zinc is copper from the copper sulphate. It is black because it is so finely divided. (1) In which tube is hydrogen evolved more rapidly? (2) Is the copper acting on the acid? (3) How do you know? (4) What technical term is used to express the action of the copper?

EXPERIMENT 9. **Preparation of Hydrogen in Quantity.**

Reference: **45.**

DISCUSSION: As has been discovered in Experiment 8, different metals and acids may be used in preparing hydrogen. Relative expense and the ease of procuring fairly pure substances may therefore determine the selection. Sulphuric acid is cheaper than hydrochloric acid. Although iron is cheaper than zinc, commercial samples usually contain considerable iron carbide, which forms with acids other gases than hydrogen. Sheet zinc contains a slight impurity of carbon, but this fact is an advantage, rather than the reverse, because the carbon acts as a catalytic agent, in the same way that copper did in Experiment 8 (*e*). The size and form of the apparatus will depend upon the amount of hydrogen to be prepared. In general a small generator is better than a large one, since it will contain less air, which must be driven out before pure hydrogen can be collected.

MATERIALS: Strips of zinc, copper sulphate solution.

DIRECTIONS: CAUTION: *Keep all flames at least five feet*

away from a hydrogen generator, as mixtures of hydrogen and air are very explosive.

Set up an apparatus like the one used in Experiment 8 (*c*). Fill three more bottles with water and invert them on the bottom of the trough. Put in the test tube 4 strips of zinc, about 5 grams, and insert the stopper. If the presence of the zinc prevents the stem of the thistle tube from entering the test tube as it should, unclamp the test tube, disconnect the delivery tube, hold the test tube horizontal, and by gently tapping it make room for the stem and insert the stopper. Then restore the apparatus to its original position. Pour into your graduate 2 c.c. of copper sulphate solution, fill it with dilute sulphuric acid to the 20 c.c. mark and pour the contents of the graduate into the test tube through the thistle tube. Upon pouring in the liquid, gas should be forced over into the bottle. If the gas does not appear, what is wrong? If this is the case, locate the trouble and remedy it at once.

Gas should begin to come over regularly into the bottle. When the bottle is a third full of gas, replace it with another bottle from the bottom of the trough, refill the first bottle with water and invert it with the others in the bottom of the trough. (1) What is the gas collected in the first bottle and rejected? After the action has proceeded for a few minutes, the gas will begin to come over faster. Feel of the outside of the test tube. (2) Why does the action increase in speed? If the action becomes so vigorous that froth rises in the test tube, wet the outside of the tube with cold water. If this is not effective, pour a little cold water down the thistle tube. (3) Why should this treatment reduce the speed of the action? When the bottle on the shelf of the trough is full, remove it as you did the bottles of oxygen and set it on the desk still covered and *still inverted.* Immediately substitute for it on the shelf one of the bottles from the bottom of the trough. Continue collecting the hydrogen till all of the bottles have been filled. After a time the hydrogen will come over more slowly. (4) To what is this due? When the rate is less than two bubbles per second, measure into your graduate 5 c.c. of concentrated sulphuric acid and pour 1 c.c. of this down the

thistle tube. When the rate of action again diminishes, pour in again 1 c.c. of acid from the graduate. Do not add more than 5 c.c. of the concentrated acid altogether, as there is danger that it will collect in the bottom of the test tube and unexpectedly becoming mixed with the water will cause a violent action. Leave the apparatus and at once study the properties of the hydrogen as directed in Experiment 10.

EXPERIMENT 10. **Properties of Hydrogen.**

Reference: **49–54.**

DISCUSSION: When a mass of gas is released into another gas, as into the air, it will rise or sink as a whole, depending on whether or not it is lighter or heavier than the gas into which it passes, just as an object thrown into water will float or sink according to its density compared to water. In addition to this movement, however, each particle of a gas has individual independent motion, which causes the gas to move against the force of gravity and to become equally distributed throughout the container which holds it. This property is called diffusion. These facts are true of all gases but they are most easily demonstrated in the case of hydrogen.

In testing for hydrogen, it will be noticed that the loudness of the explosion varies greatly on different occasions. The noise of an explosion is due to the vibrations of the air caused by the sudden production of gas or by the sudden expansion of air by heat. When hydrogen and air have become thoroughly mixed before ignition in such proportions that the oxygen present is just the proper amount to unite with all of the hydrogen, conditions are right for simultaneous action throughout the whole mass, with the instantaneous production of much heat and a consequent loud report. If, however, the amount of either the hydrogen or the air present in the mixture is too great, the amount of the other gas must be correspondingly less, and less action and less noise will result on igniting the mixture. If the gases are prevented from mixing before they are ignited, action can take place only where they meet in the process of diffusing into each other, and slow union with the phenomenon of a steady flame rather than explosion will result.

Materials: Splinters.

Directions: Stand one of the bottles of hydrogen, collected in Experiment 9, upright, remove the cover and instantly drop in a lighted match. (1) What happened? Repeat the experiment but wait fully half a minute before dropping in the match. (2) What happened? (3) Why? (4) What property of hydrogen does this experiment illustrate? (5) Why were the bottles of hydrogen prepared in Experiment 9 kept inverted?

Place a covered bottle of hydrogen mouth downward over the mouth of a bottle of air and remove the glass plate between them. After they have stood for fully five minutes, test for the presence of hydrogen in each bottle. (6) What was the result? (7) What property of hydrogen is shown by this experiment? (8) Does air possess this property? (9) Is it a property of all gases?

Thrust a lighted splinter up into an inverted bottle of hydrogen. Slowly withdraw the splinter so that it will relight at the mouth of the bottle. Look for a deposit on the inside of the bottle and feel of the neck of the bottle. (10) What took place in the bottle? (11) What was the evidence? (12) Why did no loud explosion, like those in the preceding experiments, occur? (13) What is formed when hydrogen burns in the air? (14) Does hydrogen "support combustion"? (15) What was the evidence?

QUANTITATIVE STUDIES OF CHEMICAL REACTIONS

EXPERIMENT 11. **Percentage of Oxygen in Potassium Chlorate.**

Reference: **61, 72.**

DISCUSSION: One of the most fundamental laws of chemistry is that the proportion by weight of the elements in a given compound is always the same. A knowledge of what these proportions are is, therefore, of prime importance in the study of any substance. In the case of potassium chlorate, the percentage of oxygen can be determined by heating a known weight of the compound until all the oxygen is driven off and then weighing what remains. The difference is the weight of the oxygen. In the actual experiment the weights involved are small, so that they must be determined as accurately as possible and great care must be used to lose none of the solid material. As there is no simple means of knowing when all of the oxygen has been driven off by heating, the substance must be heated, weighed, reheated and again weighed. If there is no loss of weight after the second heating, all of the oxygen was expelled on the first heating; if, however, a loss of weight appeared after the second heating, it shows that the action was not completed the first time, and successive heating and weighing must be continued until there is no further loss in weight.

MATERIALS: Potassium chlorate, pin.

DIRECTIONS: Read carefully the directions for making weighings on pages 7 to 9.

Weigh carefully a clean, dry porcelain crucible with its cover, and (1) record its weight to centigrams in the appropriate place in the tabulation given below. From a bottle of purest potassium chlorate, labeled for this experiment, transfer to the crucible with a clean, dry spatula from 1.5 to 2.0 grams

of the salt. This is about two spatulafuls. Then weigh the crucible and contents to centigrams and record the weight.

Support the covered crucible and contents on a clay triangle, resting on the ring of the ring-stand and arranged at such a height that the tip of the flame of the Bunsen burner placed underneath it will be about one inch below the bottom of the crucible. Heat the crucible in this position for about twenty minutes, then lower the ring about an inch and a half, so that the flame plays upon the bottom of the crucible, and heat it for half an hour or more. The evolution of oxygen probably will have spattered some of the potassium chlorate on the bottom of the cover. This must be removed and returned to the crucible in the following way: When the crucible has cooled, place the cover, bottom side up, on a piece of filter paper and very carefully loosen the adhering solid with a pin. Put this solid back in the crucible together with any particles which may have lodged on the paper. Now heat the covered crucible for half an hour, allow it to cool for at least half an hour, weigh it, and record the weight. Heat it again for half an hour, cool for half an hour, and again weigh it. Record all weighings.

If it has lost weight during the last heating, repeat the process until two successive weights do not disagree by more than one centigram. **(2)** Calculate the percentage of oxygen in potassium chlorate. **(3)** What is the percentage as calculated from the formula? **(4)** How great was your error?

Wt. crucible empty.
Wt. crucible and $KClO_3$.
Wt. $KClO_3$.
Wt. crucible and $KClO_3$ after first heating.
Wt. crucible and $KClO_3$ after second heating.
Wt. crucible and $KClO_3$ after third heating.
Loss of weight = weight of oxygen.
Percentage of oxygen in potassium chlorate.

EXPERIMENT 12. **The Determination of the Percentage of Chlorine in Silver Chloride.**

Reference: **74.**

Discussion: When silver chloride is heated in a stream of hydrogen, hydrochloric acid and silver are formed. By weighing the chloride in the beginning and the silver left after the reaction, the percentage of chlorine in the compound can be calculated. In the experiment described below, illuminating gas, which contains hydrogen, is used as a convenient source of hydrogen.

Materials: Silver chloride.

Directions: Weigh a clean test tube to centigrams and **(1)** record the weight. Place in it about 3 grams of silver chloride and weigh again. Support the tube in a horizontal position in a clamp placed at about one-fourth of the length of the tube from the open end. Place in the end of a rubber tube, connected with the gas cock, a piece of glass tubing about twice the length of a test tube. Turn on the gas and light it as it comes from the glass tube. Adjust the flame so that it is about 1 cm. long. Blow out the gas and insert the tube in the test tube so that the end of the former reaches almost to the silver chloride. Light the gas at the mouth of the test tube. Heat the test tube cautiously, keeping the flame in motion under the silver chloride. The test tube should not be heated hot enough to give a yellow color to the flame. As the hydrochloric acid issues from the tube it imparts a slight green color to the blue part of the flame of burning gas. It will also produce a cloud when a solution of ammonia is brought near the flame. When the reaction is completed these two tests for the gas are no longer given. When this occurs, turn off the gas, let the tube cool and weigh it. Place the silver in the bottle provided for it. What was the weight **(2)** of the silver chloride? **(3)** of the chlorine? **(4)** Calculate the percentage of chlorine in silver chloride. **(5)** What is the true percentage as calculated from the formula AgCl?

Experiment 13. **Vapor Pressure.**

Reference: **83, 89.**

Discussion: It is a matter of common knowledge that water left in an open dish will disappear, that is will form a gas or vapor which passes off into the atmosphere, will in short

evaporate. When the space above the water is enclosed, however, the vapor can not escape and as it collects in larger and larger amounts more and more returns to the liquid state, that is condenses. Eventually the rate of condensation equals the rate of evaporation and apparently no more water evaporates; vapor and liquid are said to be in equilibrium. The vapor in the enclosed space exerts a pressure on the confining walls, which pressure reaches a maximum when equilibrium between liquid and vapor, as just explained, has been attained. This maximum vapor pressure is what is referred to as *the* vapor pressure of the water.

Since water evaporates faster when hot than when cold, it follows that when equilibrium has been attained in an enclosed space condensation also must be taking place faster above hot water than above cold. But to produce more rapid condensation more vapor must be present, which in turn will cause greater pressure on the confining walls. *The* (maximum) vapor pressure of water depends, therefore, on the temperature.

The relations which exist between water and water vapor hold equally for the relation between all other liquids and their vapors. The values for these vapor pressures are different for different liquids, as would be expected from their well-known differences in rate of evaporation in the open air.

DIRECTIONS: (1) Make a drawing of the apparatus set up in the laboratory, which was prepared as follows. Each of the tubes, sealed at one end, was completely filled with mercury and then inverted in the trough of mercury. Under tube *B* was introduced a few drops of water, which rose to the surface of the mercury in the top of the tube. In a similar way ether was introduced into tube *C*. (2) What is in the space above the mercury in tube *A*? (3) What prevents the mercury from running out of the tube? (4) For what purpose is this kind of an apparatus used? (5) By what name is it known? (6) What is the length of the mercury column in tube *A*? (7) What would happen if a very little gas were let into the space above the mercury? (8) What is in the space above the mercury column in tube *B*? (9) What is the length of the mercury column in tube *B*? (10) Why is it shorter than that in tube

A? (11) What is the temperature as shown by the thermometer beside the apparatus? (12) What do you find to be the vapor pressure of water at this temperature, as expressed in terms of the height of a column of mercury? (13) What is the vapor pressure of ether at this temperature, as indicated by tube *C?*

Grasp the tube *C* with your fingers and thumb opposite the top of the column of mercury. (14) In which direction does the mercury move? (15) What is the effect of increase of temperature on the vapor pressure of a liquid?

EXPERIMENT 14. **Boyle's and Charles' Laws.**

Reference: **88–90.**

DISCUSSION: The volume of a gas increases with rise in temperature according to Charles' law, which states that the volumes of a given sample of gas at two temperatures are proportional to these temperatures expressed on the absolute scale. The absolute temperature, T, is 273 degrees plus the temperature measured on the centigrade scale, $T^\circ = 273 + t^\circ$. This law applies not only to gases like oxygen, hydrogen, and air, but also to the vapor formed from liquids such as water. In the experiment described below, the volume of a sample of air contained in a bottle over water at room temperature is first measured. It is then heated. The temperature and volume under the new conditions are recorded. A calculation is then made to determine what the volume should be according to Boyle's and Charles' laws, and this value is compared with that obtained as the result of the experiment.

The air contained in the bottle both at room temperature and at the higher temperature is mixed with water vapor, since the air is present in a bottle which also contains water. The pressure of water vapor changes with the temperature and this factor must be taken into account in the calculations. The formula which expresses the two laws is as follows:

$$\frac{pv}{p'v'} = \frac{T}{T'}$$

The value of p is determined by subtracting the pressure of water vapor at the room temperature, T, from the pressure

of the atmosphere; the value of p' is the pressure of the atmosphere minus the pressure of the water vapor at the higher temperature T'. The volume of the gas at room temperature is v, and at the higher temperature is v'. In the experiment all the factors are noted. In the calculations v' is determined by substituting the other values. If the observed value of v' agrees with the value calculated from the equation the results are a confirmation of the laws expressed by the equation.

MATERIALS: 150 c.c. bottle, large beaker.

DIRECTIONS: Arrange the apparatus as represented in Fig. 3. Place in the beaker just enough water to cover the bottle when it is completely immersed. Remove the bottle, place the beaker on a wire gauze and heat the water to 80°–85°. While the water is heating, fill the bottle to over-flowing with water at room temperature and record this temperature in the tabulation given below. Insert the stopper carrying the glass tube and press it down into place. Place the finger over the end of the tube at a and remove the stopper. Pour from the bottle about 80 c.c. of water into a graduate and note the volume of the water, which is the volume of the air in the bottle.

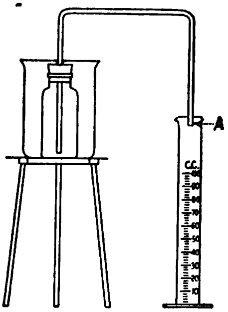

FIG. 3.

When the water in the beaker has reached 80° to 85°, fill the tube attached to the cork with water at room temperature, place the finger over the end a and replace the cork in the bottle. Remove the flame from under the beaker, put the bottle in the hot water and place it so that the water that leaves the tube at a falls into a graduate. Place a thermometer

in the hot water and stir occasionally. When water no longer falls into the cylinder note the temperature and the barometric pressure. Place the graduated cylinder under running water to cool it and note the volume of the water it contains. The sum of this volume and the original volume of the gas in the bottle is the volume of the gas at the higher temperature. Record this volume. Record the vapor pressures of water at the two temperatures involved in the experiment, as they are stated in the table of vapor pressures posted in the laboratory. (1) Calculate in the way outlined in the discussion above what the volume of the gas at the higher temperature should be. (2) By how much does this value differ from that which you found experimentally?

Temperature of room.
Temperature when water stops flowing.
Volume of air in the bottle.
Volume of water collected.
Volume of air at higher temperature.
Barometric pressure.
Vapor pressure of water at room temperature.
Vapor pressure of water at the higher temperature.

EXPERIMENT 15. **The Weight of a Liter of Oxygen.**

Reference: **80, 82, 84–88, 90.**

DISCUSSION: In finding the weight of a specified volume of gas, the direct method of weighing the gas in a flask or other container is subject to very considerable errors, unless unusual precautions are taken. These errors are due to the fact that gases are so light that the weight of a gas is very small in comparison to the weight of its container. Hence a small error in the weight of the container will make an error in the weight of the gas very large in proportion to its total weight. To avoid these difficulties the weight of the gas is often obtained indirectly, as in this experiment, by weighing the generator and contents before and after the gas has been evolved. The difference, that is the loss of weight, is the weight of the gas which has been formed and collected.

The volume of the gas is usually measured by observing

the volume of water displaced upon collecting the gas. The mere volume of a gas has no significance, however, unless the existing conditions of temperature and pressure are stated, because the volume of the same mass of gas may be greater or smaller according as the temperature is higher or lower and the pressure upon it is diminished or increased. Hence in this experiment, the temperature of the gas and the pressure under which it is confined must be determined as well as the volume which it occupies. The temperature can be read directly on a thermometer and the experiment may be so arranged that the pressure of the gas will be the same as that of the outside air. The pressure of the air, that is atmospheric pressure, is measured on a barometer. When, however, the gas is collected over water, the space above the water contains not only the gas in question but in addition water vapor, which also exerts a pressure. In other words, if the gas appears to be at atmospheric pressure, its true pressure is atmospheric pressure minus the pressure of the water vapor.

The weights of gases as usually stated are those under standard conditions, 0°C. and 760 mm. pressure, and the necessary calculation must be made by the application of the laws of Boyle and of Charles to convert the results obtained in the experiment to those which would obtain under these conditions.

MATERIALS: Potassium chlorate, manganese dioxide, copper wire.

DIRECTIONS: Thoroughly heat one spatulaful of manganese dioxide in a porcelain dish to drive off all the moisture. **(1)** What error would result if the moisture remained?

While this is heating, set up an apparatus as shown in Fig. 4. *A* is a 1 or 2 liter bottle fitted with a two-hole rubber stopper. *B* is a glass bend extending nearly to the bottom of *A*. This can be the exit tube used in Experiment 8. *C* is a right-angled bend. *D* is a dry 750 c.c. flask. *E* is a glass jet. *F* is a pinch cock. *G* is an 8-inch test tube fitted with a one hole rubber stopper. *H* is a loop of copper wire.

Mix in a mortar half a spatulaful, about 1 gram, of the dried manganese dioxide and one spatulaful, about 2 grams of potassium chlorate. Remove the test tube from the apparatus, put

into it the mixture from the mortar, suspend the test tube from the hook over the pan of a balance and determine its weight to centigrams. Before doing this read carefully the directions for weighing on pages 7 to 9. (1) Record all the data in tabular form.

Fill the jet *E* and connecting tubing *completely* with water by opening the pinch cock *F* and blowing with the mouth through the tube *C*. At the same time raise the jet and attached rubber tubing above the level of *F*. Replace the test tube in the position shown in the figure.

With the jet *E* dipped into a beaker half full of water, raise the beaker until the level of the water in the beaker is on the same level with that in the bottle *A*, then open the pinch cock *F* for several minutes to allow the pressures to become equal. (2) Why is it necessary to equalize the pressures inside and outside the bottle? Close the pinch cock and dip the jet into a clean, dry, 750 c.c. flask, as shown in the figure, which has previously been weighed to grams on the platform scales.

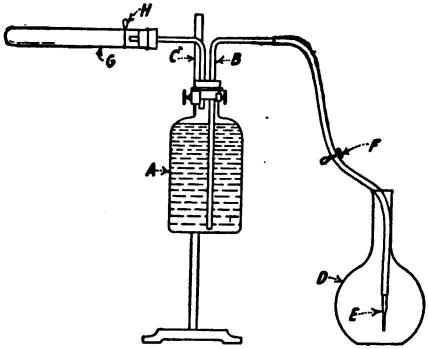

FIG. 4.

Now start heating the mixture in the test tube with a low flame and at the same time loosen the pinch cock. Water should flow out of the jet into the flask. (3) If it does not, what is wrong? Stop heating an instant, the water should cease flowing. (4) If it continues to flow, what is wrong with the arrangement of the apparatus?

When you are sure that the apparatus is correctly set up, heat the test tube more vigorously, just so that a steady stream flows from the bottle to the flask. Care must be taken at this point, *not* to heat the test tube to redness. If the heating is interrupted for any reason, be sure to keep the tip of the jet below the surface of the water in the flask. **(5)** Why? **(6)** What is replacing the water in the bottle?

Continue heating until about 600 c.c. of water has been collected in the flask. Then, with the jet still in the water, allow the test tube to cool 15 minutes to room temperature again. **(7)** What is happening to the water in the flask? **(8)** What is causing this action? Raise the flask until the level of the water in the flask corresponds with that of the water in the bottle to equalize the pressures again. Close the pinch cock.

Weigh the flask and water on the platform scales. **(9)** What is the weight of the water? **(10)** What is the volume of the gas collected? Weigh the test tube again to centigrams. **(11)** What does the loss in the weight of the tube represent? Record the temperature of the water in the flask and also the barometric pressure.

To calculate the volume of the oxygen formed, subtract from the barometric pressure the vapor pressure of water at the observed temperature of the water. (See table on page 542.) **(12)** Why? By means of Boyle's law and Charles' law calculate from the corrected pressure and from the observed temperature the volume of oxygen under standard conditions and from this value and the weight of the oxygen, the weight of a liter of oxygen. **(13)** Name all sources of error in this experiment.

EXPERIMENT 16. **Atomic Weight of Zinc or of Aluminium.**

Reference: **73–74.**

DISCUSSION: When chemical reactions take place, the proportions by weight in which given substances act with each other are always the same, as is also the proportion of the substances formed compared with the original substances. For example, the ratio between the weight of zinc used and the

weight of hydrogen evolved when the zinc reacts with acid is always the same. Furthermore it has been found that definite numbers may be assigned to all of the elements which express the proportion by weight in which they react with one another. These numbers represent the relative weights of the atoms of different elements.

In order to assign to a given element its significant number, or atomic weight, it must be experimentally determined what weight of the element is involved in chemical action with an atomic weight of some element of known value. For example if the atomic weight of element "A" is 10, and if experiment shows that 10 grams of "A" react with 100 grams of element "B," then 100 is assigned as the atomic weight of "B." In this example the assumption is made that an equal number of atoms of "A" and of "B" are involved. If, however, it is known that twice as many atoms of "B" react as atoms of "A," then the number 100 will represent twice as many atoms as the number 10 and the atomic weight of half 100 or 50 must be assigned to "B." The experiment about to be performed is an illustration of this point, for it is known that for every atom of zinc which reacts, two atoms of hydrogen are evolved; or for every atom of aluminium, three atoms of hydrogen.

In this experiment the reacting weights of zinc and hydrogen are to be found. The zinc is weighed directly. The hydrogen is too light to weigh accurately with the available apparatus, so its volume is determined under observed conditions of temperature and pressure and, after the proper corrections have been made, its weight is calculated from the known weight of a liter of hydrogen.

The atomic weight of aluminium may be determined as an alternative if desirable.

MATERIALS: Pure zinc or aluminium; concentrated, 20 per cent or 10 per cent hydrochloric acid.

DIRECTIONS: Weigh to centigrams a strip of zinc (about 2.0 grams) or a strip of aluminium (about 0.8 gram) and record the weight with the data under **1.**

Set up an apparatus as in Experiment 8 for the prepara-

tion of hydrogen, but substitute for the gas bottle a 750 c.c. flask filled with water. The flask can be supported by slipping the neck of the flask through a ring, after the flask has been filled and while it is inverted in the trough, and by clamping the ring to a stand. Be sure that the delivery tube is so placed that no gas will be lost. The water in the trough should be at about room temperature.

Put the weighed metal into the test tube, replace the stopper, and pour through the thistle tube 30 c.c. of hydrochloric acid which has been specially prepared for this experiment (20 per cent acid for the zinc, or 10 per cent for the aluminium) and to which a little copper sulphate has been added. Collect *all* the gas which comes over from the generator. If the evolution of the hydrogen becomes very slow before the last of the metal disappears, add 5 c.c. more hydrochloric acid, "concentrated" for the zinc or 20 per cent acid for the aluminium.

When all the metal has reacted, slip a glass plate over the mouth of the flask and quickly remove it from the trough, turning it right side up and being careful to lose none of the water which it still contains. Determine the volume of the water which remains in the flask, by measuring it with your graduate. Determine the total volume of the flask by filling it with water from your graduate.

(1) Record these volumes and also the temperature of the water in the trough and the barometric pressure. (2) What was the volume of the gas collected? This volume must be corrected by subtracting the volume of the acid added to the generator. (3) Why? (4) What is the corrected volume? The hydrogen left in the generator at the end of the experiment was not collected and measured. (5) Why is no correction for this amount necessary? (6) What must be subtracted from the barometric pressure in order to get the true pressure of the hydrogen? (Refer to the table on page 542 for the amount of this correction.) (7) What is the corrected pressure? (8) Calculate what the volume of the hydrogen would have been under standard conditions. One liter of hydrogen at 0° and 760 mm. pressure weights 0.090 gram. (9) What was the weight of the hydrogen collected? (10) Write in words the

proportion which you are going to use in calculating the atomic weight of the metal from your data. (11) Why do you use twice the atomic weight of hydrogen in the proportion for zinc or three times the atomic weight of hydrogen in the proportion for aluminium? (12) Substitute for the words in the above proportion the values you have obtained in this experiment and make the necessary calculations. (13) What do you find to be the atomic weight of zinc or aluminium? (14) What is the correct value? (15) How great was your error? (16) What is the ratio of your error to the correct value? (17) What do you consider the most probable source of error in your work? (18) Why?

WATER

EXPERIMENT 17. **Occurrence of Water.**

Reference: **92.**

DISCUSSION: In addition to the obvious occurrence of water in the liquid form and in the solid form, as ice and snow, water is present in many materials which on casual examination seem to be dry. In these cases the water is so intimately mixed with the other substances present that it must be separated from them before it can be recognized. Often pressure alone is sufficient to do this, that is water can be squeezed out of apparently dry objects; but in other instances the object must be heated so as first to vaporize the water, which later condenses, before its presence can be detected. There is danger, however, of being misled in these latter cases, for heating may have produced water by chemical changes in the substances of which the material is composed, instead of merely separating water which already existed as a constituent of a mixture.

Water may be present in gases as water vapor, which is invisible. (Do not confuse water vapor with fog which is made up of minute drops of liquid.) Water vapor may often be detected by cooling the gas in which it is present to such a temperature that the vapor condenses to liquid and is then recognized.

MATERIALS: Potato, wood, meat, sugar, ice, salt.

DIRECTIONS: (*a*) Cut a piece of potato with a knife and examine the blade. Repeat the experiment using some dry wood instead of the potato. (1) What is the evidence as to the presence of water in potato? (2) In the wood?

Heat a piece of wood gently in the bottom of a test tube, but do not let it char. (3) How does this experiment confirm or refute answer **2?**

Determine experimentally whether or not water is present in meat. (4) What did you do? (5) What do you conclude?

Heat a spatulaful of sugar in a test tube until it blackens. (6) What collects in the upper part of the test tube? (7) Does this prove that sugar contains water? (8) Why?

(*b*) Fill a small beaker with cracked ice, pour in water till it rises to half the height of the ice in the beaker, sprinkle into the beaker 3 spatulafuls of salt. (The presence of the salt lowers the temperature of the ice and water.) (1) What is found on the outside of the beaker after a few minutes? (2) Where did it come from? (3) Why did it appear?

EXPERIMENT 18. **Purification of Water.**

Reference: **105–106.**

DISCUSSION: The impurities in water may be present as particles of solid, or they may be in solution. To remove solid impurities, the water can be filtered, the nature of the filter depending on the size of the particles and the amount of water to be filtered. Some suspended particles are so small that they pass through all ordinary filtering materials, but often the addition of another insoluble substance will cause the fine particles to coagulate or to adhere to the substance added and then the suspended matter will be held back on the filter.

When the impurity is in solution, filtration can not be used and the water must be removed from the impurity by boiling the water and collecting the condensed vapor, that is by distillation. If the soluble impurity also distills upon boiling the water, purification becomes very difficult and special means must be resorted to for individual cases.

MATERIALS: Powdered sulphur, clay, alumina cream, copper sulphate, litmus paper.

DIRECTIONS: (*a*) Prepare samples of impure water as follows:

Sample 1. Put half a spatulaful of powdered sulphur into a test tube half full of water and shake it.

Sample 2. Grind together in a mortar half a spatulaful of clay and 30 c.c. of water.

Sample 3. Completely dissolve in an 8-inch test tube full of

water enough copper sulphate to give a distinct blue color to the solution.

Sample 4. To 30 c.c. of water in an 8-inch test tube add 5 c.c. of ammonia.

(*b*) Pour some of sample 1 into a funnel fitted with a filter paper, collecting the filtrate in a test tube. In this and the following filtration the process need be carried only far enough to show whether or not the impurity has been removed. (**1**) What has been accomplished in this case by filtration?

(*c*) Repeat experiment (*b*), using some of sample 2 instead of sample 1. (**1**) What is the appearance of the filtrate? (**2**) Why is the result different from that in experiment (*b*)? Pour 10 c.c. of sample 2 into an 8-inch test tube, add 5 c.c. of "alumina cream" (a suspension of aluminium hydroxide) and shake it thoroughly. Filter. (**3**) What is the appearance of the filtrate? (**4**) How did alumina cream accomplish the result?

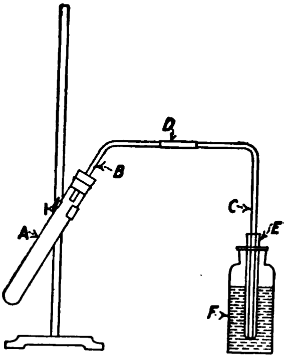

FIG. 5.

(*d*) Repeat experiment (*b*) using some of sample 3. (**1**) Was the removal of copper sulphate accomplished? (**2**) Why? (**3**) From what class of substances can water be purified by filtration? (**4**) From what class is it impossible to do this?

(*e*) Set up an apparatus for distillation as shown in Fig. 5. *A* is an 8-inch test tube provided with a one-hole stopper, *B* and *C* are glass tubes and *D* a rubber connector, *E* is a 6-inch test tube, and *F* a 250 c.c. bottle partly filled with cold water.

Put 15 c.c. of sample 3 in tube *A* and boil the solution gently, taking care that none of it spatters up through tube *B*. When a few c.c. of liquid appear in tube *E* observe the color of what has collected. (1) What is it? (2) In what physical state did it pass from tube *A* to tube *E*? (3) What is the purpose of the cold water in the bottle? (4) What is the process called which takes place in this experiment? (5) How can pure water be obtained from water containing a soluble impurity? (6) How could water fit to drink be prepared from sea water? (7) Could water be purified from an insoluble impurity by this method?

(*f*) Wet a piece of pink litmus paper with water from the tap. (1) How does pure water affect pink litmus? (2) How does solution 4 affect pink litmus paper? Repeat experiment (*e*) substituting solution 4 for solution 3. Test the distillate in tube *E* with litmus paper. (3) How does it act? (4) What does the distillate contain? (5) In what physical state did it pass from tube *A* to tube *E*? (6) From what class of substances does distillation fail to purify water?

EXPERIMENT 19. **Hydrates.**

Reference: **98–99.**

DISCUSSION: Some substances which have crystallized from water solution give off water when heated, and the original crystalline substance is again formed upon adding water to what remains after heating. If this water is present in the crystals as water, the material is obviously a mixture, a mixture of the substance and water. If such a mixture were heated, the water would be given off and the pure substance would be left unchanged. If, however, the crystals themselves were a pure substance, which was a compound of hydrogen, oxygen, and other elements, heat might decompose the compound causing the hydrogen and oxygen to form water and leaving behind some new compound, composed of the other elements. In this case heat would have brought about a chemical change. Moreover, if the hydrogen and oxygen were present as part of a compound, the law of definite proportions would apply, that is the same weight of water would always be formed from the

same weight of crystal, but if the crystals were merely a mixture of a substance with water no such definite relation would be found. Crystalline salts which behave as described above and prove to hold the hydrogen and oxygen in chemical combination are called hydrates.

MATERIALS: Rock salt, copper sulphate, potassium chlorate.

DIRECTIONS: (*a*) Grind in a dry mortar several large crystals of rock salt. (1) What evidence is there of the presence of water? Place a large crystal of the salt on a wire gauze over your burner and heat it. (2) What happened? (3) If the crystal contained within it a cavity filled with water, what would happen to this water on heating? (4) What effect would this have on the crystal? (5) Do you conclude crystals of rock salt to be pure or a mixture? (6) Why?

(*b*) Grind in a dry mortar a spatulaful of blue crystals of copper sulphate. (1) Is there evidence of water? Put the copper sulphate into an evaporating dish, on a wire gauze, supported on a ring, attached to a ring stand. Cover the dish with an inverted funnel and heat. (2) What formed on the inside of the funnel? You must watch carefully to observe this deposit, as it soon disappears again. (3) Why? (4) What change in appearance has taken place in the material in the dish? (5) Has a chemical change taken place? (6) What is the evidence? When the dish has become cool enough to handle, let a few drops of water fall upon the material which has been heated. (7) What happens? (8) Does water react chemically with the substance? (9) Do you conclude blue copper sulphate to be a hydrate or not? (10) Why?

(*c*) Find out experimentally whether or not water is given off upon heating potassium chlorate. (1) What did you do? (2) What did you find as a result? (3) Are all crystalline substances hydrates?

EXPERIMENT 20. **Efflorescence. Deliquescence.**

Reference: **100.**

DISCUSSION: As was shown in Experiment 19 some hydrates like copper sulphate may be decomposed by heating; with others, ordinary room temperature is sufficiently high to bring

about this action. Another factor besides temperature is involved, however, and must not be overlooked, namely that the anhydrous compound will take up water and change to the hydrate, if water vapor is present in sufficient concentration. Thus when a hydrate is placed in a sufficiently dry atmosphere it decomposes, giving off water. This process is called efflorescence. On the other hand, if the amount of water vapor in the air is so great that the anhydrous compound, were it formed, would immediately take up water again, no appreciable efflorescence will occur.

Another phenomenon, deliquescence, which at first sight appears to be connected with efflorescence, may be studied at this time, although it is really dependent on another principle. It will be recalled that in Experiment 15 the water vapor in the air was condensed to liquid water on the outside of a beaker containing ice. This was because the vapor pressure of water was lowered below that of the vapor in the air by cooling. When a substance is dissolved in water, the vapor pressure of the water is also diminished, and in a concentrated solution its value may become less than that of the vapor in the air, just as in the case of cooling. So on the surface of the solution condensation from the air will take place, as it did on the cold surface of the beaker. Thus a substance which is very soluble will make of itself a solution by causing condensation of water from the air and may even gather enough to become completely dissolved. Substances which cause water to be condensed upon them in this way are said to be deliquescent.

MATERIALS: Sodium carbonate, sodium sulphate, potassium chlorate, calcium chloride, sodium hydroxide.

DIRECTIONS: (*a*) Put on a glass plate a lump of crystallized sodium carbonate, upon another plate a lump of crystallized sodium sulphate and upon a third plate a few crystals of potassium chlorate. Put into a watch glass a lump of calcium chloride and into another a piece of sodium hydroxide. Label these substances and set them away on the shelf in your desk until the next exercise. Then answer the following questions. (1) Describe any changes in appearance of each. (2) Which

were efflorescent? (3) Which were deliquescent? (4) Are all substances either efflorescent or deliquescent? (5) What is the evidence? (6) How can the rate of efflorescence be increased? (7) How can efflorescence be prevented? (8) What was the source of the water which gathered on the deliquescent substances? (9) By decreasing what property of water is its vapor caused to condense? (10) What is the effect of solution on this property? (11) Do deliquescent substances form solutions? (12) What property of a substance causes it to be deliquescent?

(*b*) Put a spatulaful of dehydrated calcium chloride into a dry test tube and drop on top of it a crystal of hydrated copper sulphate. Into another dry test tube put a crystal of the copper sulphate alone. Cork the tubes and set them aside in your desk until the next exercise. Then answer the following questions. (1) What is the appearance of the copper sulphate in each tube? (2) Is the salt efflorescent in ordinary air? (3) What is the evidence? (4) What is the cause of its change in the tube containing the calcium chloride?

CHLORINE

EXPERIMENT 21. **Preparation of Chlorine.**

Reference: **112.**

DISCUSSION: The methods of preparing chlorine fall into four general classes, the electrolysis of a chloride, which is the chief industrial method, the oxidation of hydrochloric acid in which the oxygen from the oxidizing agent unites with the hydrogen of the acid and liberates the chlorine, the decomposition of a hypochlorite, and the formation of a chloride which immediately breaks up into chlorine and another chloride containing less chlorine. The last of these methods is the one commonly used for the laboratory preparation of the element.

Manganese dioxide reacts with hydrochloric acid to form manganese tetrachloride, which immediately decomposes to give chlorine and manganese dichloride. These reactions take place readily and it is usually not necessary to heat the substances. If the generation of the gas becomes very slow toward the end of the preparation, the generator may be slightly heated with a flame. Care must be taken, however, not to heat it too much, for hydrochloric acid, which is a gas dissolved in water, will be driven off and become mixed with the chlorine, if the generator becomes too hot.

A mixture of sodium chloride and sulphuric acid may be substituted for the hydrochloric acid in the preparation of chlorine. The sulphuric acid reacts with the sodium chloride and produces hydrochloric acid. This modification of the process, therefore, consists merely in making the hydrochloric acid in the generator instead of adding it already made to the generator.

MATERIALS: Manganese dioxide, concentrated hydrochloric acid.

DIRECTIONS: PRECAUTION: *Do not inhale chlorine. All experiments with chlorine must be performed in the hood.* Set

up an apparatus as shown in Fig. 6. *A* is an 8-inch test tube, *B* a 250 c.c. bottle, *C* a piece of filter paper, through the hole in which passes the delivery tube *D*. The test tube is tipped to facilitate heating it, if that becomes necessary.

Weigh out 8 grams of manganese dioxide on a filter paper, transfer it to the test tube, replace the stopper and pour in 10 c.c. of concentrated hydrochloric acid through the thistle tube. The evolution of gas should begin immediately. Place a sheet of white paper behind the bottle, so that you may see when it has become full. PRECAUTION: *Do not bend down, while watching the bottle fill, so that your head is on a level with it, because, if any chlorine has escaped from the bottle, it will lie chiefly along the floor of the hood and you may inhale it.* When the bottle is full, replace it with another and collect in all four bottles full. Heat the test tube slightly if the generation of the gas becomes too slow toward the close of the experiment. (1) What may result if the generator gets too hot? As soon as the last bottle of gas has been collected substitute for it a test tube, held in the test-tube rack, about one-third full of water. Arrange the delivery tube so that it dips into the water and continue the generation of the chlorine for three minutes. At once disconnect the apparatus and empty the contents of the generator into the large bottle in the hood provided for the purpose. (2) Write the equation for the reaction which

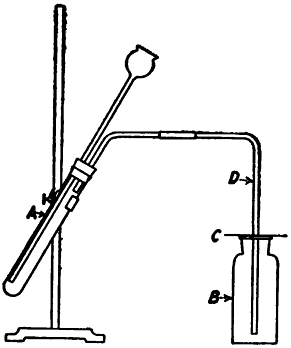

FIG. 6.

has taken place. (3) Did the manganese dioxide act as a catalytic agent as it did in the preparation of oxygen? (4) Give the reasons for your answer. (5) To which of the four general classes of preparation of chlorine named in the discussion does this method belong?

EXPERIMENT 22. **Properties of Chlorine.**

Reference: **117–130.**

DISCUSSION: Chlorine can be recognized by its odor, by its color, and by its bleaching action on certain dyes, when it is present in fairly large amounts. Small amounts of chlorine can be detected by the action on a mixture of potassium iodide and starch, but as a similar action is produced by several other substances, such as bromine, ozone, and hydrogen peroxide, this test for chlorine can be used only when these substances are known to be absent.

Chlorine will unite with elements of widely different chemical nature, such as phosphorus, copper, and hydrogen, but the difficulties of demonstrating all of these reactions are too great to make the experiments suitable for a large elementary class. The reactions of some of the metals, however, can easily be shown, as well as the behavior of chlorine toward the compounds of carbon.

The reaction of chlorine with water illustrates a new type of reaction, one in which part of the chlorine forms one compound and part forms another, hydrochloric acid and hypochlorous acid. A new principle is also exemplified here, namely that of an incomplete reaction, where there exist together in equilibrium both some of the original substances and some of the products of the reaction. The reaction between chlorine and water is important because it is involved in the process of bleaching.

MATERIALS: Iron wool, copper wire, wax taper, colored cotton cloth, newspaper, fountain pen ink, litmus paper, starch, potassium iodide solution.

DIRECTIONS: *Perform these experiments in the hood.* (*a*) (1) What is the color of chlorine? (2) Describe its odor. (3) Is it heavier or lighter than air? (4) What is the evidence?

(5) Why was it not collected by displacement of water, as was hydrogen?

(*b*) Ignite a wad of iron wool in the burner and quickly thrust it into a bottle of chlorine. (1) Describe the result. (2) What is the name of the substance formed? (3) Write the equation. (4) Assuming that other metals behave like iron, make a statement concerning the action of chlorine with metals in general.

(*c*) Thrust a blazing wax taper into a bottle of chlorine. The taper is composed of compounds of carbon and hydrogen. (1) What is deposited on the sides of the bottle? (2) What element does not unite directly with chlorine? Blow across the mouth of a bottle of concentrated hydrochloric acid. (3) What did you observe? (4) Blow across, not into, the mouth of the bottle into which the burning taper was thrust. PRECAUTION: *Be careful not to inhale the gas from the bottle.* (5) What does this test show was formed by the action of the chlorine on the taper?

(*d*) Fasten together on the ends of two copper wires two sets of strips each consisting of a piece of colored cotton cloth, a piece of litmus paper, and a piece of printed newspaper, upon which has been made a cross with fountain pen ink and one with lead pencil. (The "lead" of a lead pencil and printers' ink are composed of forms of carbon.) Thoroughly wet one of the sets, and suspend each set in a bottle of chlorine. (1) State what happened to the cloth, the litmus paper, the printers' ink, the fountain pen ink, and lead pencil mark in each case. (2) Did the method of preparing the chlorine produce dry or moist chlorine? (3) Why? (4) Judging by the relative action on the two sets of strips, what do you believe would have been the action on the dry set, had the chlorine been dried before using it? (5) Is dry chlorine a bleaching agent? (6) Is water? (7) What was the bleaching agent in this experiment? (8) What elementary substance is not bleached by chlorine in the presence of water?

(*e*) Grind in your mortar a lump of starch the size of a small pea, transfer the starch to a small test tube, fill the tube half full of water, and heat it to boiling. Add 5 drops of

potassium iodide solution and then wet a strip of filter paper with the liquid. Hold the piece of filter paper in the bottle of chlorine in which the dry cloth and paper were suspended in (*d*). (1) What happens to the starch iodide mixture on the paper? (2) What is a delicate test for chlorine? (3) What common substances must be absent in order to make this test trustworthy?

(*f*) Put a drop of dilute hydrochloric acid, of dilute sulphuric acid, and of dilute nitric acid on each of three pieces of blue litmus paper. (1) What is the action of acids on blue litmus paper? (2) What is a test for an acid? Dip the end of a glass rod into the water into which you passed chlorine in Experiment 21 and touch a piece of blue litmus paper with it. (3) What change in color takes place *immediately?* (4) What kind of a compound or compounds does the water contain? (5) What are the names of the substances formed by the action of chlorine on water? (6) Write the equation for the reaction. Cautiously smell the solution. (7) Does it still contain chlorine? (8) Did the reaction, the equation of which you have just written, proceed to completion? (9) What is the evidence? (10) What substances are present in "chlorine water"? (11) Which of the substances is the bleaching agent in "chlorine water"? (12) What are your reasons for ascribing the action to this compound rather than to the other substances present?

EXPERIMENT 23. **Other Means of Preparing Chlorine.**

Reference: **116.**

DISCUSSION: When hydrochloric acid is treated with a suitable oxidizing agent, such as potassium permanganate, the oxygen from the oxidizing agent unites with the hydrogen of the hydrochloric acid and chlorine is liberated. These methods are often very convenient but are more expensive than the method involving the use of manganese dioxide.

When a hypochlorite is treated with hydrochloric acid, hypochlorous acid is formed from the hypochlorite. It was shown in Experiment 22 *f* that the reaction between water and chlorine to form hydrochloric acid and hypochlorous acid was

incomplete. Incomplete reactions are reversible, that is, the products of the reaction partially interact in turn and produce again some of the original substances. Thus chlorine and water form hydrochloric and hypochlorous acids and these will react again to form chlorine and water. Both reactions proceed simultaneously, so that at any given moment all four substances are present. When now a hypochlorite is treated with an excess of hydrochloric acid both hypochlorous acid and hydrochloric acid will be present, and, as just stated, they will react to give chlorine.

Bleaching powder is a complex salt from which both hydrochloric and hypochlorous acids are produced when it is treated with many different acids, such for example as sulphuric acid. It is thus a convenient substance from which to prepare chlorine.

MATERIALS: Potassium permanganate, concentrated hydrochloric acid, sodium hypochlorite solution, bleaching powder, cotton and silk cloths.

DIRECTIONS: (*a*) Put five crystals of potassium permanganate in a test tube, add not more than 5 c.c. of concentrated hydrochloric acid and heat the tube slightly. Cautiously smell at the mouth of the tube. (1) What was formed? (2) To what class of substances does potassium permanganate belong? (3) State the general method of preparing chlorine of which this experiment is an example.

(*b*) To 5 c.c. of sodium hypochlorite solution in a test tube add 5 c.c. of concentrated hydrochloric acid and heat the tube slightly. (1) Was chlorine evolved? (2) How did you test for it? (3) What was formed in the first place by the action of the acid on the hypochlorite? (4) Write the equation for the reaction between this product and hydrochloric acid. (5) To what reaction studied in Experiment 22 is this related and how? (6) What term is applied to a pair of such reactions?

(*c*) Put a spatulaful of bleaching powder into an evaporating dish and add enough water to make it into a thick paste. Add 10 drops of dilute sulphuric acid and cover one end of a strip of colored cloth with the mixture. After it has stood for 5 minutes, remove the cloth and wash it under the tap. (1)

What has happened to the cloth? (2) What produced the action? (3) What was the source of this substance?

(*d*) On a piece of white cotton cloth make a spot about the size of a dime with fountain pen ink. Spread the spotted part of the cloth over an evaporating dish and slowly pour drop by drop a few cubic centimeters of sodium hypochlorite on the spot. Next pour on in the same way a few cubic centimeters of dilute hydrochloric acid. Wash the cloth in plenty of water. (1) What was the action on the ink? (2) What practical application can be made of these solutions? (3) What would be the result of removing ink from colored cloth by this method?

Repeat the experiment just described, using a piece of thin silk cloth instead of cotton cloth. (4) What was the result? (5) Draw a conclusion as to the range of usefulness of this means of removing ink stains.

HYDROCHLORIC ACID

EXPERIMENT 24. **Preparation of Hydrochloric Acid.**

Reference: **138.**

DISCUSSION: The study of the action of sulphuric acid upon sodium chloride is important, since this is the reaction which is commonly made use of in preparing hydrochloric acid, since it is an example of a very general method for the preparation of acids (namely, the action of sulphuric acid on a salt), and since it affords an excellent illustration of double decomposition.

It will be recalled that in the preparation of hydrogen by the action of an acid on a metal in Experiment 8 the metal took the place of the hydrogen of the acid and the hydrogen thus liberated, having nothing to unite with, escaped as a gas. If, however, a compound of a metal had been used instead of the uncombined metal, the other element or elements of the compound would have been left when the metal replaced the hydrogen of the acid. This element or these elements thus set free would become available to unite with the hydrogen just displaced and to form a compound. Thus the metal and hydrogen would have changed places, that is a reaction of "double decomposition" would have taken place. In this case, therefore, the sodium of the sodium chloride will displace the hydrogen of the sulphuric acid, which in turn will unite with the available chlorine and produce hydrochloric acid.

If this reaction is to be used to prepare hydrochloric acid economically, the reverse action, namely hydrochloric acid and sodium sulphate forming sodium chloride and sulphuric acid must be prevented. Since, however, hydrochloric acid is a gas, it may be allowed to escape as fast as formed and not enough will remain to allow the reversal.

As sulphuric acid contains two atoms of hydrogen in each molecule, the sodium may replace one or both of the hydrogen

atoms forming either sodium hydrogen sulphate or disodium sulphate. At the temperature of the laboratory preparation the former reaction takes place, at higher temperatures the latter.

MATERIALS: Sodium chloride.

DIRECTIONS: *Perform this experiment in the hood.* Measure out into an evaporating dish 8 c.c. of water and add to it 10 c.c. of concentrated sulphuric acid. (Do not add the water to the acid.) While the solution is cooling set up an apparatus like the one for the preparation of chlorine, Experiment 19. Put 10 grams of sodium chloride into the test tube and pour the acid down the thistle tube. When the salt has become thoroughly moistened with the acid, heat the test tube with a low flame, holding the burner in your hand. To determine when the bottle has been filled, hold a piece of wet blue litmus paper at its mouth. Do not consider the bottle full when the first test is obtained but continue generating the gas until it overflows in some quantity. Collect four bottles full and cover them with glass plates.

Immediately empty the generator into the bottle provided for the purpose. (**1**) Write the equation for the reaction which took place in the generator. (**2**) For the reaction which would have taken place at a higher temperature. (**3**) To what class of reactions does this preparation of hydrochloric acid belong? (**4**) What elements changed places? (**5**) What property of hydrochloric acid made the completion of the reaction possible?

EXPERIMENT 25. **Properties of Hydrochloric Acid.**

Reference: **139–146.**

DISCUSSION: A cloud is always composed of minute particles of liquid or solid. A gas or vapor is always clear and when colorless is invisible. In common parlance a cloud of water, like the clouds in the sky or like fog, is sometimes spoken of as vapor. This is, strictly speaking, incorrect, as the cloud is really made up of minute drops of liquid water, water vapor being invisible.

When a gas "fumes" in the air, that is when a cloud appears upon the gas coming in contact with the air, small

drops of solution are formed. This occurs when the gas is so soluble that a solution can be produced the vapor pressure of which is less than the pressure of the water vapor existing in the air. Condensation then takes place and drops of liquid are formed. This condensation depends upon the same principle as does deliquescence which was discussed in Experiment 20 and should be here reviewed.

For the recognition of hydrochloric acid in solution two independent tests are necessary. The solution must be shown to contain an acid and also a chloride. Many substances are acids, many other substances are chlorides, hydrochloric acid alone is at the same time both an acid and a chloride.

In testing for a chloride the principle of elimination is made use of. The formation of a white precipitate upon the addition of silver nitrate indicates the presence of any of a certain group of substances; the action of nitric acid upon the precipitate reduces the possibilities to a certain few; and the action of ammonia indicates the individual among these.

MATERIALS: Splinter, litmus paper, concentrated hydrochloric acid, solutions of silver nitrate, sodium chloride, potassium chloride, sodium carbonate, potassium bromide.

DIRECTIONS: (*a*) (1) What is the color of hydrochloric acid? (2) Describe its odor. (3) Is it heavier or lighter than air? (4) What is the evidence?

(*b*) Into a bottle of the gas collected in Experiment 24 thrust a lighted splinter. (1) What happened? (2) From what other gases which you have studied can hydrochloric acid be distinguished by this experiment?

(*c*) Invert in a pneumatic trough one of the bottles of hydrochloric acid. Keep the bottle covered until its mouth is under the water. Shake the bottle slightly and hold it so that its mouth is just below the surface of the water in the trough. Observe the height of the water inside the bottle, slip a glass plate over the mouth of the bottle and remove it together with the water which it contains. Test the water in the bottle with blue litmus paper. (1) What does the bottle contain? (2) What is the evidence? (3) When the bottle was inverted in the trough, what was the level of the water inside

compared to that outside? (4) Explain why this was so. (5) Is hydrochloric acid soluble or insoluble in water? (6) What was the gas remaining in the bottle? (7) How did it get there? (8) Can hydrochloric acid be collected by displacement of water? (9) Why?

(*d*) Blow across, not into, the mouth of a bottle of gas. (1) What appeared? (2) What was it composed of? (3) What was the source of the water? (4) Why did it condense? (5) Would an insoluble gas "fume" in the moist air? (6) Why? (7) To substantiate your answer, name an insoluble gas you have studied and state whether or not it "fumes."

(*e*) Wet a piece of filter paper with a solution of ammonia and drop the paper into a bottle of hydrochloric acid gas. (1) What appeared? (2) Is it a solid, liquid, or gas? (3) What is the name of the substance? (4) Write the equation for the reaction by which it was formed.

(*f*) (1) What does the reagent bottle marked "hydrochloric acid" contain? Blow across the mouth of a bottle of concentrated hydrochloric acid and also across the mouth of a bottle of dilute hydrochloric acid. (2) What happened in each case? (3) Explain the reason for the difference in behavior.

(*g*) (1) Hydrochloric acid may be regarded as the chloride of what element? To three test tubes add 1 c.c. of dilute hydrochloric acid, 1 c.c. of sodium chloride solution, and 1 c.c. of potassium chloride solution, respectively, and then fill each a third full of water. Dip into each a piece of blue litmus paper. (2) Record the results. (3) How can a solution of hydrochloric acid be distinguished from solutions of other chlorides? To each of the three solutions add 10 drops of silver nitrate solution, then 2 c.c. of nitric acid and finally 5 c.c. of ammonium hydroxide. Shake the tubes and be sure that enough ammonium hydroxide has been added to give a distinct odor of ammonia at the mouth of each tube. (4) Record what occurred on the addition of each reagent to each tube. To two test tubes add 1 c.c. of sodium carbonate solution and 1 c.c. of potassium bromide solution respectively. To each add 10 drops of silver nitrate solution. (5) What happened in each case? (6) Is the appearance of a white precipitate

upon the addition of silver nitrate a conclusive test for a chloride? (7) Why? Add 2 c.c. of nitric acid to each test tube. (8) What happened in each case? (9) How does the addition of nitric acid distinguish the precipitate produced from a chloride by silver nitrate from that produced from a carbonate by silver nitrate? To the test tube which contained the potassium bromide add 5 c.c. of ammonium hydroxide or enough to give a distinct odor of ammonia. (10) What happened? (11) In what respect does a bromide behave differently from a chloride? (12) State the complete test for a chloride. (13) Why is it necessary to add nitric acid? (14) Why ammonium hydroxide?

(*h*) (1) In what experiment have you studied the action of hydrochloric acid on a metal? (2) What was formed? (3) Write the equation for the reaction.

EXPERIMENT 26. **Preparation of Lead Chloride from Lead Oxide.**

Reference: **142.**

DISCUSSION: Hydrochloric acid reacts with oxides and forms chlorides. The preparation of lead chloride in this way is easy to carry out because the compound is only slightly soluble in cold water. The equation for the reaction is as follows:

$$PbO + 2HCl = PbCl_2 + H_2O$$

MATERIALS: Lead oxide, concentrated hydrochloric acid.

DIRECTIONS: Place 4 grams of lead oxide in a beaker and add to it 300 c.c. of water and 30 c.c. of concentrated hydrochloric acid. Boil the solution for about 5 minutes and then pour it off from the undissolved solid into a second beaker. Let the solution cool to room temperature. Filter off the crystals and set the paper aside until it is dry. Weigh the lead chloride on the paper, using a piece of filter paper of the same size in the other pan to counter-balance the weight of the original paper. (1) Record the weight of the compound. (2) Describe its appearance.

EXPERIMENT 27. **The Quantitative Determination of the Hydrogen and Chlorine in Hydrochloric Acid.**

Reference: **142–143.**

DISCUSSION: When zinc reacts with hydrochloric acid each atom of the metal replaces two atoms of hydrogen. This fact can be used to determine the amount of hydrogen in a given weight of the acid by treating the latter with a weighed amount of zinc. When reaction is complete the metal is weighed again. The loss in weight is a measure of the hydrogen, since 65.4 grams of zinc dissolve when 2.016 grams of hydrogen are set free.

The chlorine in a known weight of hydrochloric acid can be determined by adding to the solution of the acid a solution of known concentration of silver nitrate, as long as silver chloride is precipitated. From the volume of the solution of silver nitrate used and from the concentration of the latter (number of grams $AgNO_3$ per cubic centimeter) can be calculated the weight of silver nitrate and the weight of chlorine in the acid.

MATERIALS: Sheet zinc, silver nitrate, concentrated hydrochloric acid, 10 c.c. graduate.

DIRECTIONS: (*a*) Measure accurately from a 10 c.c. graduate 5 c.c. of pure concentrated hydrochloric acid. Pour the acid into a graduated cylinder and dilute with water to 50 c.c. In order to avoid adding too much water, it is advisable to fill the cylinder up to between 45 and 50 c.c. and add the last portion from a glass tube used as a pipette. Stir the solution with a glass rod until it is thoroughly mixed.

Exactly 10 c.c. of this solution is to be used to determine the chlorine and 40 c.c. to determine the hydrogen. Measure accurately from the graduate 40 c.c. of the solution into a beaker, and place in it a clean piece of sheet zinc about 4 cm. square that has been weighed to centigrams. (1) Record the weight. Warm the acid until reaction begins and then set it aside to use in part *c*.

(*b*) Weigh to centigrams about 2.5 grams of silver nitrate, transfer it to a graduated cylinder, and add water to the 50 c.c. mark. Stir until the solution is uniform.

Add to exactly 10 c.c. of the hydrochloric acid already prepared about 50 c.c. of water and then a dilute solution of sodium hydroxide until the solution is alkaline to litmus paper. From the cylinder add to the solution about 25 c.c. of the silver nitrate and stir. Add more of the latter cautiously until the colorless milky solution changes to a light yellow-brown color. (1) Record the volume used. As long as the solution contains a chloride white silver chloride is formed. When the chloride has been precipitated, silver oxide, which is brown, is formed as the result of the action of silver nitrate on the sodium hydroxide present. Calculate (2) from the weight of silver nitrate dissolved in 50 c.c. the weight of silver nitrate in 1 c.c.; (3) from the number of cubic centimeters of silver nitrate used and from the weight in 1 c.c., the weight used to react with the hydrochloric acid; (4) from the fact that 1 $AgNO_3$ precipitates 1 Cl as AgCl, calculate the weight of chlorine precipitated. This weight of chlorine was contained in 10 c.c. Calculate (5) the weight in the 50 c.c. which contained the 5 c.c. of concentrated hydrochloric acid.

(*c*) Returning to the zinc and hydrochloric acid solution in part *a*, warm the beaker if the reaction has not ceased. When the evolution of hydrogen is very slow, the solution can be boiled gently. Cool, and if the gas is no longer evolved, remove the metal, wash it in running water, and dry it on a clean towel. In about 5 minutes weigh it. Record the weight of zinc under *a* 1. (1) From the loss of weight of the zinc and from the fact that 65.4 grams of zinc liberate 2.016 grams of hydrogen, calculate the weight of hydrogen evolved. This amount of hydrogen was contained in 40 c.c. of the acid analyzed. (2) Calculate the amount in 50 c.c. The chlorine in this amount of acid was calculated in *b* 5 above. (3) From these figures calculate the weight of hydrochloric acid contained in the 50 c.c. (4) From the weights of the acid, hydrogen, and chlorine calculate the percentages of chlorine and of hydrogen in hydrochloric acid.

(*d*) Weigh a graduated cylinder, fill it to the 50 c.c. mark with concentrated hydrochloric acid and weigh again. (1) Calculate the weight of 1 c.c. of concentrated hydrochloric

acid. (2) Calculate the weight of 5 c.c. of the acid. From this weight and the amount of pure acid found in *c* 3 above, calculate (3) the percentage of hydrochloric acid in concentrated hydrochloric acid.

HYDROGEN PEROXIDE

EXPERIMENT 28. **Preparation and Properties of Hydrogen Peroxide.**

Reference: **165–167.**

DISCUSSION: Hydrogen peroxide can be prepared by the double decomposition of a metallic peroxide and an acid. It is a rather unstable compound, easily giving up oxygen with the liberation of energy. It acts thus as an oxidizing agent and exhibits those properties which would be predicted from this fact, such as a bleaching action on certain colored substances, and the ability to set free iodine from potassium iodide.

In the test for hydrogen peroxide with potassium dichromate in acid solution the colored substance formed is more soluble in ether than in water. Hence in making the test, if the solution is shaken with a little ether, the colored compound is extracted by it and concentrated into smaller volume, where it is more easily detected. The rate of decomposition of hydrogen peroxide with the evolution of oxygen gas is greatly increased by the presence of any fine powder, like manganese dioxide, which acts as a catalytic agent.

MATERIALS: Solution of hydrogen peroxide, potassium dichromate, ether, barium peroxide, manganese dioxide, cochineal, potassium iodide, starch.

DIRECTIONS: (*a*) To become familiar with the test for hydrogen peroxide, add to a test tube half full of water 10 drops of dilute sulphuric acid, 2 c.c. of potassium dichromate solution, a layer of ether 2 cm. thick and finally 3 drops of hydrogen peroxide solution. Shake the mixture. (1) What color is the ether layer? (2) What is the function of the ether in the test?

(*b*) To 10 c.c. of water in a small beaker add 5 c.c. of dilute sulphuric acid and enough ice so that pieces will rest on the bottom of the beaker. Slowly sprinkle in a spatulaful of

barium peroxide, stir the mixture and filter it. Test the filtrate for hydrogen peroxide. (1) What did you do in making the test? (2) What did you conclude from the test? (3) Write the equation for the formation of the hydrogen peroxide. (4) Why was the ice used?

(*c*) Into 10 c.c. of hydrogen peroxide solution in a test tube sprinkle a quarter of a spatulaful of manganese dioxide. If a vigorous evolution of gas does not immediately result, heat the mixture. (1) What in all probability is this gas? Verify your prediction by making a test. (2) How did you test the gas? (3) What did you find it to be? (4) Write the equation for its formation. (5) What was the function of the manganese dioxide? (6) In what experiment was it used for a similar purpose?

(*d*) Prepare some starch iodide paper as you did in Experiment 22 *e* and put a few drops of hydrogen peroxide solution on it. (1) What occurred? (2) The presence of what element is indicated by the color? (3) Write the equation for the reaction by which this element was formed. (4) What other substance which you have studied produces this action on starch iodide paper? (5) What property of hydrogen peroxide is illustrated by this action?

(*e*) To 5 c.c. of dilute cochineal solution in a test tube add 5 c.c. of hydrogen peroxide solution and heat. (1) What took place? (2) Because of what chemical property of hydrogen peroxide did this action take place?

EXPERIMENT 29. **The Quantitative Analysis of Hydrogen Peroxide.**

Reference: **166–167.**

DISCUSSION: When a solution of hydrogen peroxide is treated with potassium permanganate, $KMnO_4$, oxygen is set free, one-half of which comes from the peroxide and one-half from the permanganate. The volume of the gas liberated in this way from a known volume of a solution of hydrogen peroxide is a measure of the strength of the latter.

MATERIALS: Potassium permanganate, solution of hydrogen peroxide.

Directions: Arrange an 8-inch test tube with a thistle tube and a delivery tube to collect a gas over water. Add to the tube a pinch of potassium permanganate and 10 c.c. of water. Insert the stopper and place the end of the delivery tube under a gas bottle filled with water. Pour into the test tube 10 c.c. of a solution of hydrogen peroxide and then 10 c.c. of water. When gas ceases to be evolved, cover the mouth of the bottle, remove it from the trough and measure (1) the volume of the water it contains. Fill the bottle with water and measure (2) its volume. The difference (3) is the volume of the gas that was collected. To get the volume of the oxygen (4) subtract 20 c.c. from (3). (5) Calculate what volume of gas is generated when 1 c.c. of hydrogen peroxide is decomposed by potassium permanganate.

PROPERTIES OF LIQUIDS AND SOLIDS

EXPERIMENT 30. **Determination of Density.**

Reference: **175–176.**

DISCUSSION: The density of a solid or a liquid is defined as the weight in grams of 1 c.c. of the substance. In order to determine the density of a substance it is necessary to know the weight and volume of a sample of it. If the substance is a liquid its density can be determined readily by weighing a measuring vessel, such as a graduated cylinder, placing in it the liquid, the volume of which is noted, and then weighing the two. The weight of 1 c.c. of the liquid can then be determined by calculation. In order to obtain a fair degree of accuracy, it is advisable to weigh as much as 50 c.c., if the platform scales are to be used.

If the density of a solid is to be determined, its volume is found out by immersing it in a known volume of a liquid in which it does not dissolve, and noting the sum of the volumes of the liquid and the solid. This can be done by placing a known weight of the solid in a known volume of the liquid contained in a graduated cylinder. The increase in volume after the solid has been added is the volume of the latter. Water is used most conveniently in the case of metals and other substances which are insoluble in water. In the case of salts that dissolve in water, benzene, kerosene, or carbon tetrachloride can be used.

MATERIALS: Concentrated hydrochloric acid, iron nails, sodium chloride, benzene.

DIRECTIONS: (*a*) Weigh on the platform scales to decigrams your graduated cylinder, which should be dry. Fill it exactly to the 50 c.c. mark. In order to do this pour in water until it is nearly up to the mark and add the rest drop by drop from a piece of glass tubing full of water, over one end of

which the finger is held. If the pressure of the finger is reduced and a little air is allowed to enter the upper part of the tube the water can be made to run out of the tube drop by drop. (1) Weigh the cylinder and the water. (2) What is the error, if any, in the graduation of the cylinder?

Pour out the water, dry the cylinder, fill it exactly to the 50 c.c. mark with concentrated hydrochloric acid, and weigh again. (3) Calculate the density of the acid.

(*b*) Place about 40 c.c. of water in the graduated cylinder and note accurately to tenths of a cubic centimeter its volume. Slide into the cylinder about 20 grams of nails, which have been weighed to decigrams on the platform scales. Read to 0.1 c.c. the volume of the water and iron. (1) Record all data. (2) What does the difference between this volume and that of the water represent? (3) Calculate the density of iron.

(*c*) Carry out a similar experiment using 20 c.c. of benzene and 20 grams of sodium chloride. (1) Record the results. (2) Why is benzene used instead of water? (3) What is the relation between density and specific gravity?

EXPERIMENT 31. **Determination of Specific Heat.**

Reference: **177.**

DISCUSSION: The specific heat of a substance is the amount of heat required to raise the temperature of 1 gram of the substance 1 degree centigrade. In determining specific heat use is made of the following facts: The specific heat of water is 1, that is, 1 calorie raises the temperature of 1 gram of water 1 degree. The heat given off by a hot body when its temperature falls 1 degree is equal in amount to that required to raise its temperature 1 degree.

Directions are given below for the determination of the specific heat of a solid. A known weight of brass is heated in water to a definite temperature. It is then transferred to a known weight of water at room temperature. The rise in temperature is noted. The product of the number of degrees rise in temperature and the weight of the water is evidently the number of calories given off by the brass. This figure divided by the weight of the brass gives the number of calories

given off by each gram of brass. The figure obtained in this way divided by the number of degrees through which the temperature of the brass fell is the number of calories given off when 1 gram of brass drops 1 degree in temperature, that is, the specific heat of brass.

In actual practice some of the heat given up by the brass is lost to the surrounding air and to the glass of the beaker and the thermometer. As a consequence, the rise in the temperature of the water will not be an exact measure of the heat lost by the brass. A correction is made, accordingly, in a way which can be understood from the following considerations. When two equal weights of water at different temperatures are mixed, the temperature of the mixture is just half way between the temperatures of the two portions before they were mixed, provided no heat is lost to the vessels containing the water. Thus, if 50 c.c. of water at 20° is mixed with 50 c.c. of water at 30°, the temperature after mixing would be 25° if no heat were lost. If heat is lost, however, the difference between the observed temperature and the mean of the two temperatures is a measure of this heat.

MATERIALS: One 200 gram brass weight attached to a string.

DIRECTIONS: (*a*) Fill a 500 c.c. beaker about two-thirds full of water, place in it the brass weight, and heat the water to about 75°. Measure into a 200 c.c. beaker 100 c.c. of water at room temperature. (**1**) Record the temperature of the water estimating the temperature to 0.1 degree. Remove the beaker containing the brass from the burner, stir the water slowly with the thermometer, read the temperature (preferably when the column of mercury stands at a degree mark) and immediately transfer the brass by means of the string to the small beaker, and (**2**) note the time. Cool the thermometer to room temperature by placing it in water at this temperature, and insert it into the small beaker containing the brass. Observe the thermometer as the temperature rises slowly. (**3**) Record the highest temperature reached and the time when this occurs. From the two time observations it will be seen how many minutes are necessary for all the heat to be transferred from the brass. This time (3 to 5 minutes) will be required in the second part

of the experiment. For convenience it will be referred to later as *T*.

(*b*) To determine to what extent the loss of heat to the surroundings affects the rise in temperature proceed as follows: Measure into the small beaker used before 50 c.c. of water at room temperature. (**1**) Record the temperature of the water to 0.1 degree. Place 50 c.c. of water in a small flask and heat it until its temperature is approximately twice as many degrees above the temperature of the cold water as the observed rise in part (*a*) above. (**2**) Read the temperature of the warm water to 0.1 degree, pour it at once into the 50 c.c. of cold water in the beaker and (**3**) note the time. Cool the thermometer to room temperature by immersing it in water at this temperature, and put it into the beaker in which the two portions of water were mixed. (**4**) Record the temperature after the time *T* (see (*a*) above) has elapsed. If there had been no loss of heat from the water to the air, the beaker, and the thermometer, the final temperature would have been just half way between the temperatures of the cold and the hot water (the average of the two). The difference between this temperature and that actually observed is the error introduced as the result of the loss. This difference (**5**) should be added to the rise in temperature observed in (*a*) above.

(*c*) (**1**) From the corrected rise in temperature found in (*a*) calculate the specific heat of brass in the way outlined in the discussion.

(**2**) What is meant by the specific heat of a substance? (**3**) Where did most of the heat of the brass go? (**4**) Where did the remainder go? (**5**) Why was all possible speed used in transferring the brass from the beaker? (**6**) Why was the time observed? (**8**) How was the error of heat loss corrected for?

EXPERIMENT 32. **The Effect of Pressure on the Boiling Point of Water.**

Reference: **178.**

DISCUSSION: Boiling is the formation of vapor within a liquid, as distinguished from evaporation from the surface only. A liquid boils when the pressure of its vapor just exceeds

the pressure exerted upon its surface. Under these conditions a bubble of vapor can form under the surface of the liquid. Thus, when a liquid is heated in a vessel open to the air, the pressure of the latter must be overcome before it boils. If a part of the air is withdrawn from the vessel, in any way, the air exerts less pressure and, as a consequence, the liquid boils at a lower temperature. This is due to the fact that a smaller vapor pressure, attained at a lower temperature, is now enough to exceed the diminished pressure of the air upon the liquid.

The experiment described below illustrates this fact in a simple way. Water is boiled in a vessel connected to a tube which dips under water in a second vessel (Fig. 7). As the liquid boils the water vapor formed drives out most of the air which escapes through the tube. When the air has been driven out, the tube is closed and the source of heat removed from the boiling water. Since the apparatus is now closed, the only pressure upon the surface of the water is that of the small amount of air present, plus the vapor already there. As this latter condenses upon cooling, boiling continues for some time at the reduced pressure, while the temperature is falling.

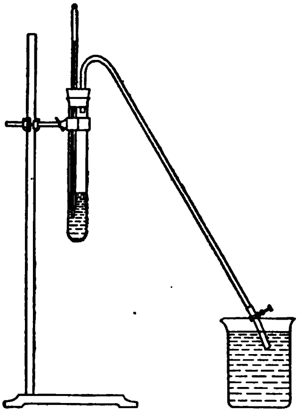

FIG. 7.

MATERIALS: Rubber tubing.

DIRECTIONS: Arrange an apparatus as represented in Fig. 7. Use an 8-inch test tube and a glass tube about 18 inches long. The bulb of the thermometer should be under the surface of the water so that the temperature of the latter can be observed. (In determining the boiling point of a liquid the thermometer is ordinarily placed in the vapor of the boiling liquid, when heat is constantly supplied from an outside source.)

In order to aid boiling place a few pieces of broken glass or tile in the test tube and add about 20 c.c. of water. Insert the stopper carrying the delivery tube which is fitted with a short piece of rubber tubing and a pinch cock. Open the latter and heat the water to boiling. Observe closely the change in size of the bubbles that escape through the water in the beaker as the boiling continues. (1) State what occurs and give a reason for the observed facts.

When the steam makes a hissing sound as it condenses in the beaker (2) note the temperature of the boiling water. Cautiously heat the test tube with a burner held in one hand and close the pinch cock with the other hand. Remove the flame immediately, and raise the beaker so that the end of the glass tube is well covered by the cold water. Observe the thermometer as the water continues to boil. (3) Note the lowest temperature observed when the water is boiling. Tap the tube lightly to assist in the formation of bubbles of vapor.

When boiling ceases open the pinch cock under the water. (4) What happened? (5) Why? Was there any pressure exerted by air on the boiling liquid when the system was closed? How could you calculate the value of this pressure from the observations made? (6) Did the apparatus contain any air when closed? (7) What is the evidence? (8) How does boiling differ from surface evaporation? (9) What conditions must exist for boiling to take place? (10) Ordinarily boiling is produced by the increase of what pressure? (11) How is this accomplished? (12) By the decrease of what pressure may boiling be brought about? (13) How can water be made to boil below 100°?

EXPERIMENT 33. **Heat of Vaporization.**

Reference: **180.**

DISCUSSION: When a liquid passes into a vapor, heat is required to effect the change. The heat required to convert 1 gram of a substance from the liquid to the vapor state is called the heat of vaporization.

When a liquid is boiled over a flame the heat required is obtained from the burning gas. When a liquid evaporates of itself the heat is supplied by the surrounding air and by the liquid; as a consequence the temperature of the latter falls. If the conditions are such that the liquid evaporates rapidly, a relatively large amount of heat must be supplied in a short time, and the temperature of the liquid falls to a greater extent than when the evaporation takes place slowly and heat can be taken up from the surrounding air.

The extent to which the change in temperature takes place is determined by the rapidity of evaporation, the heat of vaporization of the liquid, and the rate at which the material can take heat from the surroundings.

MATERIALS: Piece of string.

DIRECTIONS: (*a*) Cut from a piece of filter paper a strip about 2 cm. wide by 10 cm. long. Wrap the paper around the bulb of the thermometer and tie it in place with a piece of string. (1) Note the temperature of the air. Dip the end of the thermometer in ether, hang it up and observe the temperature. (2) Record the lowest temperature reached.

(*b*) Dry the paper or replace it by a new piece and repeat the experiment using carbon disulphide. (1) Record the temperature.

(*c*) Repeat the experiment using alcohol. (1) Record the temperature.

(*d*) Repeat the experiment using water. (1) Record the temperature.

(2) What property of a liquid determines the rate at which it evaporates at a given temperature, provided it receives a constant supply of heat sufficient for the evaporation? (3) If two liquids are evaporating at the same rate what determines the relative amounts of heat absorbed in the two cases? (4)

The heat of vaporization of ether is 90 calories and that of alcohol 206 calories. What conclusion as to their relative vapor pressures can you draw from these facts and the results obtained in the experiment? (5) Upon heating a liquid why does the temperature cease to rise when it begins to boil, although the heating is continued?

EXPERIMENT 34. **Distillation of a Mixture of Two Liquids.** Reference: **181.**

DISCUSSION: A liquid possesses a definite boiling point at a fixed pressure, and a definite density at a fixed pressure and temperature. The density of a homogeneous mixture of two liquids varies with the proportions of the two substances present. When such a mixture is heated to boiling the lower boiling liquid does not distill off first in the pure condition at its own boiling point, but a mixture is obtained, the composition of which varies as the distillation proceeds. The distillate always contains a larger percentage of the more volatile liquid than is present in the boiling mixture. As the distillation proceeds the proportion of the higher boiling liquid thus increases. The experiment described below is designed to illustrate the

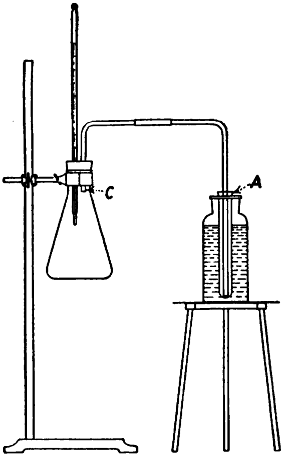

FIG. 8.

behavior of such a mixture when it is slowly distilled. The liquids used are methyl alcohol (wood alcohol), which boils at 66°, and water, which boils at 100°.

MATERIALS: 250 c.c. flask, methyl alcohol, rubber band.

DIRECTIONS: Set up the apparatus represented in Fig. 8. A test tube, *A*, placed in cold water in a gas bottle serves to collect the distillate. (1) Weigh a graduated cylinder to decigrams. Place in the graduate 15 c.c. of methyl alcohol, note the volume to 0.1 c.c. and weigh again. (2) Calculate the density of the alcohol. Pour the alcohol into the flask. Wash out the cylinder with water and then add 30 c.c. of water to the alcohol. Shake the mixture so that it is uniform. Dry the cylinder. Pour into it the mixture. Note the volume accurately and weigh. (3) Calculate the density of the mixture.

Pour 2 or 3 drops of the mixture on a glass plate and apply a lighted match. (4) Does it burn? Pour the mixture into the flask and place a rubber band around the tube which is to receive the distillate in such a position that it will serve to show when approximately 15 c.c. of liquid have been collected. To do this put 15 c.c. of water into the test tube, place the tube in position in the bottle and mark the level of the water by the position of the band. Empty and dry the test tube and place it again in the bottle which contains cold water. Arrange the apparatus as indicated in the diagram. As the flask is heated in the way described below, (5) note the temperature recorded on the thermometer when the liquid first drops back from the tube at *C* and also when the distillation is stopped. Heat the flask cautiously with a flame. When the liquid begins to boil remove the flame, count 5 seconds, heat again till the liquid boils (about 1 second), and remove the flame. Continue heating cautiously in this way until 15 c.c. of the distillate have been collected. (6) Determine the density of the distillate, and return it to the test tube.

Cool the contents of the flask under running water and (7) determine its density.

Pour a few drops of the distillate on a glass plate and determine whether or not it will burn. Test the residue in the flask in the same way. (8) What were the results? (9) What

do you conclude about the relative amounts of alcohol in the distillate and in the residue?

Pour the distillate into the flask, replace the stopper and thermometer and (10) determine the boiling point of the liquid. (11) Arrange in tabular form the boiling points, the densities, and the percentages of alcohol of the following: methyl alcohol, water, mixture, distillate, and residue.

From the values of the densities obtained, the percentage of methyl alcohol in the mixtures can be found from the following table:

METHYL ALCOHOL AND WATER

Density	.983	.972	.961	.946	.929	.909	.887	.863	.838	.810
Per cent of alcohol	10	20	30	40	50	60	70	80	90	100

(12) What is the density of a mixture of 1 volume of methyl alcohol and 2 volumes of water as calculated by finding the sum of one-third the density of the alcohol and two-thirds of the density of the water? (13) What was the observed density? (14) What occurred when the liquids were mixed? (15) Can the composition be calculated directly from the density? (16) How did you find the relation of composition to density? (17) What is the relation of the composition of the distillate to that of the boiling mixture? (18) How does the composition of the mixture change during distillation? (19) Why does the boiling temperature rise?

EXPERIMENT 35. The Determination of a Freezing Point.

Reference: **187.**

DISCUSSION: The freezing point of a pure liquid is the temperature at which it changes to a solid. The melting point of a pure solid is the temperature at which it changes to a liquid. These temperatures are the same, for any one substance. If heat is constantly supplied to a solid it rises in temperature until the melting point is reached. The temperature then remains constant as heat is supplied, since the added heat is used in con-

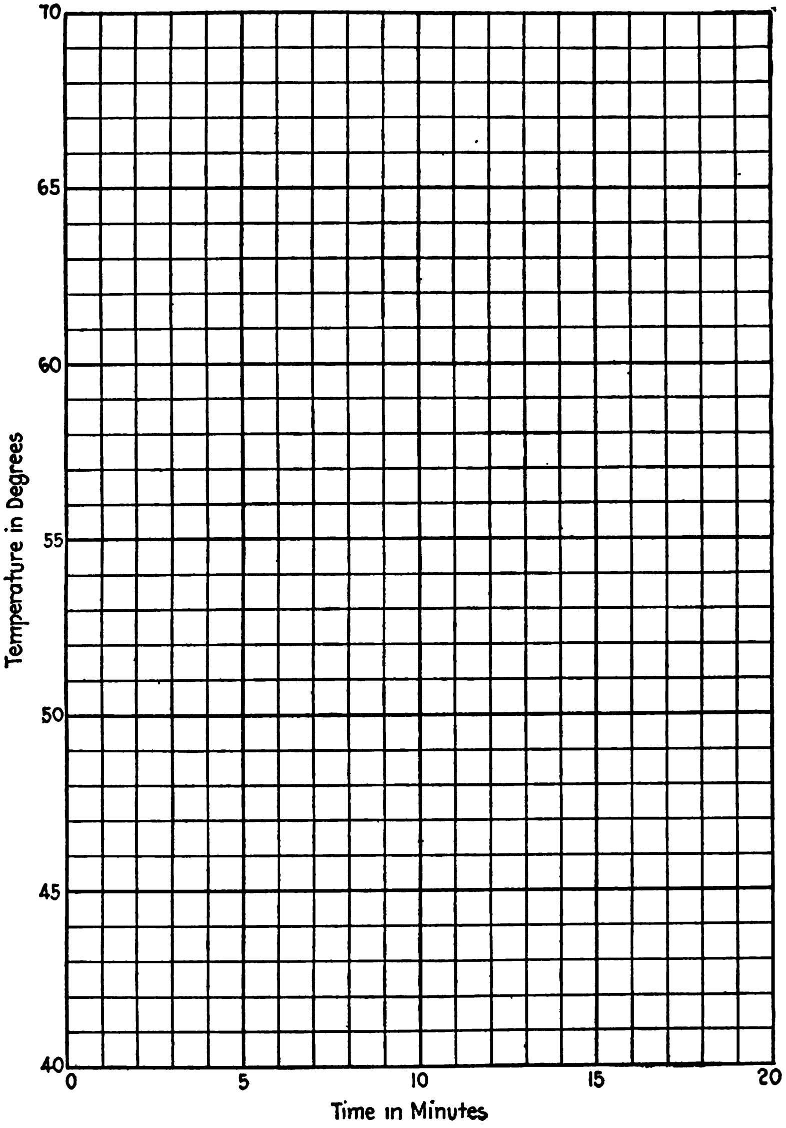
70
65
60
55
50
45
40
Temperature in Degrees
0
5
10
15
20
Time in Minutes

FIG. 9.

verting the solid into the liquid. When the solid has melted, the temperature of the liquid again begins to rise as the heat is supplied. Conversely, when heat is withdrawn from a liquid it falls in temperature until the freezing point is reached. The temperature remains constant at this point as crystallization takes place, because the formation of crystals from the liquid occurs with the evolution of heat.

The change which takes place when a liquid solidifies is illustrated by the experiment described below. The freezing point can also be determined in the way illustrated in the next experiment.

MATERIALS: Sodium thiosulphate, $Na_2S_2O_3,5H_2O$.

DIRECTIONS: Put approximately 10 grams of sodium thiosulphate in a dry test tube and place the latter in hot water (about 70°) in a beaker until the solid has melted. Remove the test tube from the water, place the thermometer in it, and stir slowly. Read the temperature at intervals of a minute. When the thermometer falls to about 55° drop into the tube a small crystal of sodium thiosulphate. A little of the solid is added to prevent supercooling (see next experiment). Continue the stirring and record the temperatures at intervals of a minute for 10 minutes. (1) Plot the results on Fig. 9.

(2) What occurred in the liquid during the time that it did not change in temperature? (3) Why is heat lost to the surroundings continually throughout the experiment? (4) What is the evidence that heat is produced during part of the experiment? (5) Which part was this? (6) What was the source of the heat? (7) What is the temperature called at which this action occurs? (8) What other change will occur at the same temperature? (9) What is this temperature called?

EXPERIMENT 36. **Supercooling.**

Reference: **189.**

DISCUSSION: Many liquids can be cooled below the temperature at which they freeze, provided the conditions exist which prevent the formation of the solid form. When a trace of the solid is added to such a supercooled liquid, crystallization takes place. As the change of a liquid to a solid is accom-

panied by the evolution of heat, the temperature of the liquid rises to that of the melting point and remains constant as the solidification proceeds.

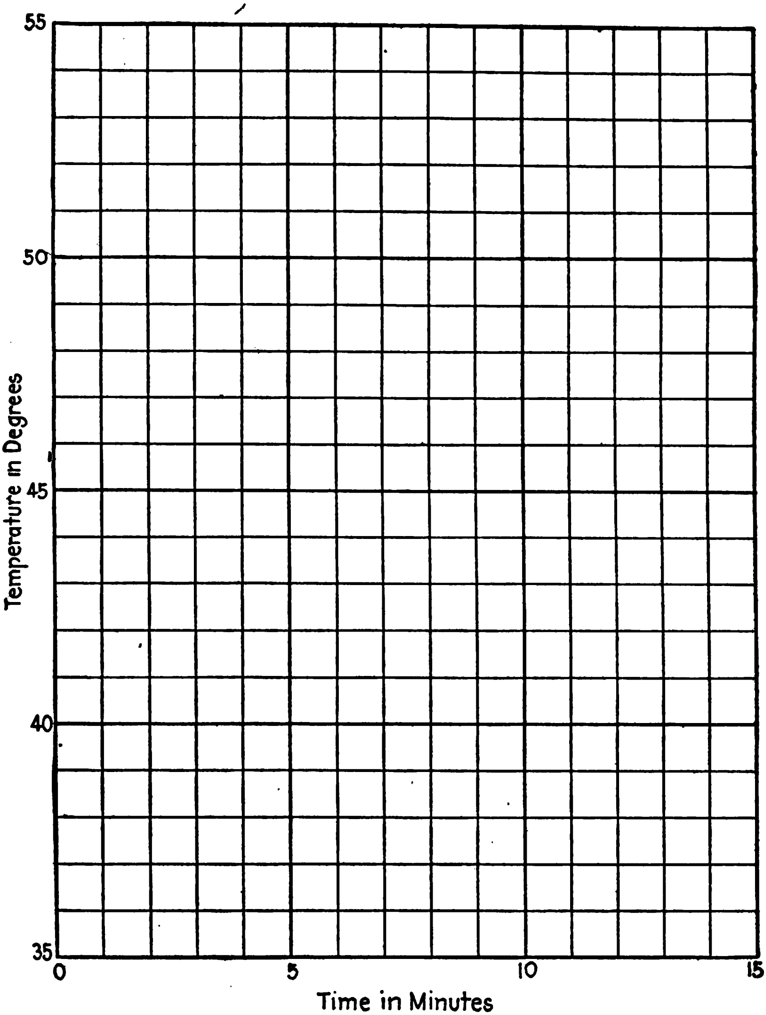

FIG. 10.

MATERIALS: Sodium thiosulphate.

DIRECTIONS: Melt approximately 10 grams of sodium thio-

sulphate in a dry test tube placed in hot water in a beaker at about 70°. Care should be taken that the solid particles of the substance do not adhere to the walls of the tube above the liquid. Remove the test tube from the water, insert a thermometer, and cool the tube under running water until the temperature of the liquid is approximately 40°. Drop a small crystal of sodium thiosulphate into the tube, stir, and note the temperature each minute for 10 minutes.

(1) Plot the results on Fig. 10. (2) At what degree did the temperature remain constant for a period? (3) What was taking place during this period? (4) How does this temperature compare with that of constancy in Experiment 35? (5) What is this temperature called? (6) In what condition is the liquid said to be when it is below this temperature? (7) What precaution must be observed to maintain this condition? (8) What was the source of heat when the temperature rose?

CARBON AND ITS COMPOUNDS

EXPERIMENT 37. **Properties of Carbon.**

Reference: **191–197.**

DISCUSSION: Carbon does not possess a single set of properties as do hydrogen and chlorine, but three very different sets. A given sample is called diamond, graphite, or amorphous carbon, according to which set of properties it possesses. The specimens of amorphous carbon which are familiar have all been formed by the decomposition of compounds of carbon, often mixed with other substances, and so are seldom pure. The differences in the methods of formation and the presence of the impurities make the amorphous carbon from different sources appear to have different properties, as in the case of coal, charcoal, and soot; but when the pure carbon is extracted from these substances, it is all found to be the same.

The physical properties of these allotropic forms vary more widely than do the chemical properties and it is the peculiar physical properties of diamond and of graphite which make these substances of commercial value.

MATERIALS: Powdered graphite, lumps of charcoal, powdered wood charcoal, animal charcoal, vinegar, hydrogen sulphide solution, lead oxide.

DIRECTIONS: (*a*) Rub a little graphite between your thumb and forefinger. (1) Describe the feeling. (2) What use is made of graphite dependent on the property which causes this feeling?

Rub a bit of graphite across a white paper. (3) What was produced? (4) What common use of graphite depends upon this property?

Make a small hole in a lump of charcoal and fill it with some powdered graphite. Holding the burner in your hand, let the flame play directly on the hole. (5) Which ignites more readily, the graphite or the charcoal?

(*b*) Put a small lump of charcoal, not more than a centimeter cube, into water. (1) Does it sink or float? Hold the lump of charcoal with the tongs in the flame until it is red hot and quickly put it in the water again. (2) Does it now sink or float? (3) What is the cause of the difference in behavior? (4) What does this experiment suggest as to the structure of a piece of charcoal?

(*c*) Fill a test tube a quarter full of animal charcoal, add 10 c.c. of vinegar, shake the test tube until the charcoal has become moistened throughout, and then heat it to boiling. Filter into a test tube and compare the color of the filtrate with some of the original vinegar. (1) What change in appearance has been produced? (2) Was this a physical or a chemical change? (3) To what physical property of the charcoal was the action due?

Repeat the experiment with the substitution of 10 c.c. of hydrogen sulphide solution for the vinegar. Compare the odor of the filtrate with that of the original solution. (4) What change has the charcoal produced? (5) What has become of the hydrogen sulphide? (6) What uses of charcoal do these experiments suggest?

(*d*) Mix in your mortar one-quarter of a spatulaful of lead oxide and the same amount of powdered wood charcoal. Put some of the mixture in a hole in a lump of charcoal and heat it with the blow pipe. A little preliminary practice with the blow pipe may be necessary, which may be carried out as follows: First, with your mouth closed, puff out your cheeks and breathe through your nose several times, till you can do this freely. Now put the blow pipe in your mouth and repeat the performance, keeping your cheeks puffed out. The air in your mouth will become exhausted only slowly and may be replenished by exhaling into your mouth from time to time, while still breathing through your nose. After a little practice you will be able to blow a continuous stream of air through the blow pipe. Slip the blow-pipe tube down the tube of your burner, light the gas and turn it down till the flame is about half an inch high. Hold the blow-pipe in your mouth, steadying it with the right hand and letting the tip rest in the crotch

of the blow-pipe tube. Hold the piece of charcoal in the left hand and direct against it the flame produced by blowing into the tube. Heat the mixture of lead oxide and charcoal for two or three minutes, then shake it out on your desk and examine it. (1) What substance do you find? (2) Write the equation for the reaction by which it was formed. (3) As what kind of an agent has the charcoal acted? (4) In what commercial processes are similar reactions made use of?

EXPERIMENT 38. **Preparation of Carbon Dioxide.**

Reference: **200.**

DISCUSSION: Of the various methods for the formation of carbon dioxide, which will be taken up later in Experiment 40, the most convenient for laboratory preparation is the action of an acid on a carbonate. The progress of the reaction depends upon the instability of carbonic acid, which breaks up into water and carbon dioxide. Substances are chosen, therefore, which will react to form carbonic acid by double decomposition and these are, as just stated, a carbonate and an acid. If a slow steady stream of gas is desired, an insoluble carbonate, usually calcium carbonate, is selected, since a soluble carbonate reacts too rapidly. The acid employed must be one which will form a soluble compound with the metal of the carbonate, for if an insoluble compound were formed, it would coat over the solid carbonate and prevent further access of the acid.

MATERIALS: Marble, concentrated hydrochloric acid.

DIRECTIONS: (*a*) Use the same apparatus as for the preparation of hydrogen in Experiment 8. Fill the generator about one-third full of lumps of marble, slipping the pieces in while the tube is inclined, so that they will not crack it. To 20 c.c. of water in a graduate add 20 c.c. of concentrated hydrochloric acid and pour through the thistle tube enough of this mixture to cover completely the marble. Collect three bottles of carbon dioxide as you have collected gases over water before. (1) Which side up should the filled bottles be kept? (2) Why? (3) By the spontaneous decomposition of what substance is the carbon dioxide formed? (4) Write the equations for the

two reactions taking place in this preparation. (5) To what class of reactions does the first belong? (6) The second? (7) Why does the first take place to completion? (8) Why does the second? (9) Write the equations for the reactions which would have taken place if sodium carbonate had been used instead of calcium carbonate. (10) If sulphuric acid had been used instead of hydrochloric acid. (11) Why is calcium carbonate preferred over sodium carbonate? (12) Why is a hydrochloric acid used instead of sulphuric acid? (See table of solubilities, page 543.)

(*b*) Pour the liquid out of the generator, put in more marble if necessary, disconnect the delivery tube at the rubber connector and substitute for it a tube passing to the bottom of an 8-inch test tube through a two-hole rubber stopper. Into the other hole of the stopper fit a glass tube, bent at a right angle and barely passing through the stopper. Connect this tube by means of a rubber connector to a second right-angled tube passing into a second test tube. Fill both test tubes one third full of water, pour acid into the generator as in (*a*), and allow the action to proceed for at least 10 minutes. Save the contents of both test tubes for Experiment 39.

EXPERIMENT 39. **Properties of Carbon Dioxide.**

Reference: **201–205.**

DISCUSSION: Carbon dioxide may be used to illustrate the behavior of a gas heavier than air, because, being odorless, it produces no disagreeable results if it escapes. Many other gases, as chlorine and sulphur dioxide, are much heavier but are not suitable for experiments on density. Likewise the action of carbon dioxide on burning matter is not a unique property of this gas but this phenomenon can be most conveniently shown with carbon dioxide because it is so easily formed and entirely inoffensive.

In the test for carbon dioxide calcium carbonate is produced. It is obvious that in using this test care must be taken to prevent acid from coming in contact with the reagent, for the acid would act on the calcium carbonate. A fine spray from the acid used in the production of carbon dioxide often acts this way unless precaution is taken.

Materials: Splinter, lime water, silver nitrate solution, litmus paper.

Directions: (*a*) (1) What is the color of carbon dioxide? (2) What is its odor?

(*b*) Thrust a blazing splinter into a bottle of carbon dioxide. (1) What occurred? (2) What other gas which you have prepared acted similarly? (3) Why is carbon dioxide to be preferred to this gas as a fire extinguisher?

(*c*) Bend a splinter at a right angle, light an end of it and lower it into a bottle of air. Immediately pour into the bottle a bottleful of carbon dioxide, holding the bottles mouth to mouth, and pouring as if the carbon dioxide were a liquid. (1) What occurred? (2) Is carbon dioxide heavier or lighter than air? (3) What is the evidence? (4) What other gases which you have prepared have a similar density compared with air?

(*d*) Into a bottle of carbon dioxide pour 5 c.c. of lime water (a solution of calcium hydroxide) and shake the bottle. (1) What appeared? (2) What is the name of the compound? (3) Write the equation for the reaction for its formation. (4) What is the test for carbon dioxide? Add 5 c.c. of hydrochloric acid to the bottle. (5) What occurred? (6) What precaution must be taken in testing for carbon dioxide?

(*e*) Test with litmus paper the solution contained in the second of the test tubes through which carbon dioxide was bubbled in Experiment 38 *b*. Add 5 c.c. of lime water to the solution. (1) What was the action on the paper? (2) With the lime water? (3) What compound does the solution contain? (4) Write the equation for its formation. (5) Does the reverse of this reaction take place? (6) What is the evidence? (7) Explain how it is that carbon dioxide reacts with water and yet can be collected over water.

Test for a chloride in the water contained in the first test tube in Experiment 38 *b*. (8) What did you do to make the test? (9) What was the result? (10) What do you conclude the water contains? (11) How did it get there? (12) Why did it not pass on into the second test tube? (13) Why was it necessary to have this tube as part of the apparatus?

EXPERIMENT 40. **Formation of Carbon Dioxide by Various Means.**

Reference: **200.**

DISCUSSION: As was shown in Experiment 38, carbon dioxide is formed from the spontaneous decomposition of carbonic acid (hydrogen carbonate) which in turn is formed by the action of an acid on a carbonate. Carbonates other than hydrogen carbonate decompose giving carbon dioxide and an oxide, but they must be heated above room temperature. The heating of limestone to make lime is a notable example of this action.

The burning of carbon in air is an obvious method of formation, as is also the complete oxidation of compounds of carbon, either by combustion or natural processes of slow oxidation. Some compounds of carbon also form carbon dioxide by decomposition in the process of fermentation, which is brought about by the presence of micro-organisms.

MATERIALS: Sodium carbonate, lime water, magnesium carbonate, marble, charcoal, paper, candle, "Karo Syrup," yeast cake, copper wire.

DIRECTIONS: (*a*) To half a spatulaful of sodium carbonate in a test tube add 5 c.c. of dilute sulphuric acid. Hold in the mouth of the test tube, with a steady hand, a glass rod which has been dipped into lime water and upon the end of which a drop of the lime water is hanging. (1) How did the drop change in appearance? (2) What was formed? (3) What gas was found to be present? (4) If the drop shows no change, what is the probable cause for the failure of the experiment? (5) The action between what other carbonate and acid have you studied? (6) Describe the test for a carbonate.

(*b*) Heat in a small test tube half a spatulaful of manganese carbonate and then test for carbon dioxide as you did in experiment (*a*). (1) What is the evidence as to the decomposition of the manganese carbonate? (2) Write the reaction. Repeat the experiment substituting a lump of calcium carbonate for the manganese carbonate. (3) Is there evidence of decomposition? (4) In what commercial process is calcium carbonate decomposed by heat? (5) Why is this not used as a laboratory method for preparation?

(*c*) Hold a piece of charcoal with the tongs in the flame until it glows and then quickly thrust it into a bottle of air for a minute. Test the gas in the bottle for carbon dioxide. (1) How did you make the test? (2) What was the result? (3) Write the equation for the burning of the charcoal.

Drop a piece of burning paper into a bottle of air and test for carbon dioxide. (4) Of what element is paper a compound? (5) What is formed when it burns? (6) What is the evidence?

Fasten a candle to a stout copper wire and after lighting it thrust it into a bottle of air for a minute. Test for carbon dioxide in the bottle. Seek evidence of the formation of two compounds. (7) What do you conclude they are? (8) By what observation or experiment did you recognize their presence? (9) Of what elements do you conclude a candle to be a compound?

Blow through a tube into a test tube half full of lime water. (10) What is one of the constituents of the exhaled breath? (11) What is the evidence of this? (12) By what kind of a reaction was this constituent of the breath formed? (13) What was the source of the necessary oxygen? (14) What is the evidence of the production of heat in the formation of the compound? (15) Name two kinds of action taking place in nature in which carbon dioxide is formed without the phenomenon of combustion.

(*d*) Mix in a 300 c.c. Erlenmeyer flask 20 c.c. of "Karo Syrup" and 150 c.c. of water. Grind in a mortar a quarter of a yeast cake with 10 c.c. of water and pour the mixture into the flask. Fit into the flask a one-hole rubber stopper through which passes a tube bent at right angles and connect this tube by means of a rubber connector with another right-angled tube which dips into 50 c.c. of lime water in a 250 c.c. bottle. Set the whole apparatus away in your desk until the next exercise, when you should answer the following questions. (1) What has been formed in the bottle of lime water? (2) What is shown to be one of the products of fermentation? (3) Is this a reaction of oxidation? (4) What brought about the fermentation?

EXPERIMENT 41. **Formation of Carbonates.**

Reference: **204.**

DISCUSSION: As appears from the preceding experiments, calcium carbonate is formed when carbon dioxide is passed into a solution of calcium hydroxide. Similar reactions occur between carbon dioxide and the hydroxides of other metals. The reaction may be regarded as taking place in two steps, first the production of carbonic acid from the carbon dioxide and the water of the solution and then a double decomposition between the acid and the hydroxide forming the carbonate and water. When a solution of some other compound of the metal is substituted for the hydroxide, as for example the chloride, an acid will be formed by the double decomposition instead of water. But since, as has been shown, an acid always reacts with a carbonate, these two could not be formed and exist together. In other words a carbonate can not be prepared by passing carbon dioxide into a solution, if the other product of the reaction is an acid.

Acid carbonates are compounds of both a metal and hydrogen in combination with the carbonate radical. They are formed by the action of an excess of carbonic acid on a normal carbonate. This reaction, however, can be reversed by boiling off the carbonic acid as carbon dioxide and water.

MATERIALS: Solutions of calcium chloride and of calcium hydroxide, marble.

DIRECTIONS: (*a*) Bubble carbon dioxide from the generator you used in Experiment 38 *b* into 10 c.c. of calcium chloride solution. **(1)** How does the result differ from that obtained when carbon dioxide is passed into calcium hydroxide? **(2)** Explain the cause of this difference. **(3)** How can calcium carbonate be prepared from calcium chloride? Try the method which you suggest. **(4)** What leads you to believe that the substance which you have obtained is calcium carbonate?

(*b*) Pass carbon dioxide which has been purified from hydrochloric acid, as in Experiment 38 *b*, into a test tube one-third full of lime water, until the precipitate first formed disappears. **(1)** What was the precipitate? **(2)** Into what compound was

it converted? (3) Write equations for all the reactions taking place in the test tube.

Boil the solution in the test tube until a distinct change is noticeable. (4) What appeared? (5) Write the equation for the reaction which took place. (6) What is the relation of this reaction to the one in which the precipitate disappeared? (7) What is such a pair of reactions called? (8) Which is more soluble, calcium carbonate or calcium acid carbonate?

EXPERIMENT 42. **Coal.**

Reference: **220.**

DISCUSSION: Coal is the product of the slow decomposition of vegetable material in the absence of oxygen. Since this decomposition is never complete, coal is found to be a mixture, different samples varying in their composition. Upon heating coal many of these undecomposed compounds, which are hydrocarbons, are given off, some as permanent gases and others as easily condensed substances, which appear as a tar. Much of the nitrogen and sulphur in combination in the original vegetable material are converted into ammonia and hydrogen sulphide, which are evolved with the other gases when the coal is heated. The residue, which is called coke, consists of uncombined carbon and inorganic material, the latter being left as ash when the coke is burned in the air.

MATERIALS: Bituminous coal, litmus paper.

DIRECTIONS: Half fill a test tube with powdered bituminous coal and heat it vigorously. Smell the fumes, and when they are coming off vigorously, try to ignite them. Test the liquid which collects near the mouth of the test tube with pink litmus paper. (1) Describe the appearance and odor of the fumes. (2) Of what class of compounds are they chiefly composed? (3) Are they combustible? (4) Describe the appearance of the deposit at the mouth of the tube. (5) What is it called? (6) What was the action upon litmus paper? (7) What is thus proved to be given off from the coal? (8) What is the residue called which is left upon heating the coal? (9) What industrial process is illustrated by this experiment?

EXPERIMENT 43. **Flame and the Bunsen Burner.**

Reference: **230–231.**

DISCUSSION: The distinction between amount of heat and intensity of heat, that is, temperature, must be kept clearly in mind. The same amount of heat is produced when the same amount of gas is burned no matter what kind of flame is produced, but the temperature of the flame will be higher when the combustion takes place in a smaller space and when there is less inert gas to be heated. Thus a given amount of gas burning as a luminous flame produces the same amount of heat as it does burning as a Bunsen flame, but the latter is hotter, as the space where the burning takes place is smaller. Likewise the same amount of heat is produced when gas burns in the air as is produced when it burns in pure oxygen, but the latter flame is hotter since no heat is consumed, as in the former case, in raising the temperature of the inert nitrogen.

The preliminary admixture of air to the gas, affected by the Bunsen burner, serves the double purpose of increasing the temperature of the flame and of preventing the deposit of soot on objects heated. Soot is the carbon formed by the decomposition of the hydrocarbons of the gas where there is not enough oxygen present to consume it. In the luminous flame the only source of oxygen is the surrounding air, as no oxygen exists in the center of the flame to consume the carbon; but in the Bunsen flame the air drawn in at the bottom of the burner furnishes oxygen within the flame for this purpose and so no soot is formed.

Although the preliminary addition of air to the gas is advantageous, the amount must be such that the mixture will not ignite in the tube of the burner itself. In a properly regulated burner a combustible mixture of gas and air is rising through the tube, but it does not reach its kindling temperature because the flow of the cold mixture upward offsets the conduction of heat from the flame downward. When the latter is more rapid the mixture becomes ignited and the gas burns at the base of the burner. The rate of flow of the mixture, however, is not the only factor involved, for the addition of

too much air may cause the flame to "strike back," since the additional amount of oxygen produces a mixture in the tube which is explosive and which is ignited by the gas burning at the top of the burner. If something, like a metal grid for example, is placed in the top of the tube of the burner to conduct away the heat of the flame and thus prevent the mixture of air and gas from being raised in temperature, a more combustible mixture, one with more air in it, can be used. This will enhance the effect produced by an ordinary Bunsen burner and a flame of still higher temperature will be obtained. The Meker burner is constructed on this principle.

In the Bunsen flame three distinct regions exist. Directly above the burner is a cone-shaped space filled with the mixture which has just emerged from the burner but which has not yet been heated to the kindling temperature and is not burning. This is surrounded by gas burning in the air drawn in through the burner, but here the combustion is incomplete, because there is an insufficiency of oxygen and an excess of gas. This part of the flame is known as the reducing cone, because a decomposable oxide introduced into it will have its oxygen taken away to aid in the combustion. This reducing cone is in turn surrounded by a region where an excess of oxygen derived from the air exists and where an oxidizable substance may take on oxygen. This part of the flame is the oxidizing cone.

MATERIALS: Fine copper gauze, pin, copper wire, Meker burner.

DIRECTIONS: (*a*) Take apart a Bunsen burner. Three separate pieces are obtainable. (1) Make drawings of the parts of the burner.

(*b*) Put the burner together again, close the holes in the bottom of it, light the gas, and hold a glass rod in the upper part of the flame. (1) What is deposited upon it? (2) Was this substance present in the unburned gas as it issued from the cock? (3) Where did it come from? (4) What becomes of it when no object upon which it may be deposited is held in the flame? (5) Why does this action not take place within the flame?

Admit air into the burner and again hold the rod in the flame. (6) How does the result differ from that previously obtained? (7) Why?

(*c*) Turn the gas on fully, close the air inlet and measure the height of the flame by holding a ruler beside it. Open the air inlet and again measure the height of the flame. (1) Record the heights. Hold a platinum wire at the tip of the green cone of the Bunsen flame and then, after closing the air inlet, in the luminous flame. (2) Which flame was the hotter? (3) What is the evidence? (4) What is the cause? (5) Was more heat produced in one flame than in the other? (6) Why? (7) What are the advantages of the Bunsen flame over the luminous flame?

(*d*) (1) Make a drawing of the Bunsen flame.

Hold a piece of glass tubing at an angle of 45 degrees with the lower end in the cone immediately above the top of the burner and apply a lighted match to the other end of the tube. (2) What occurred? (3) What does this prove about the composition of this part of the flame?

Stick a pin through a match close to the head. Turn off the gas and hang the match head upwards in the tube of the burner by means of the pin. Turn the gas on fully and light it. (4) What happened to the match? (5) What does this prove about the temperature of this part of the flame?

Hold a bright stout piece of copper wire in the tip of the flame. (6) What color does it become? (7) What is the substance which produces the color? (8) What does this prove as to the composition of this upper part of the flame? (9) What is this part of the flame called?

(10) What is the color of the part of the flame directly outside the innermost cone? Hold the end of the copper wire, which you have just heated, in this part of the flame. (11) What change in color do you observe? (12) What reaction is taking place on the surface of the wire? (13) What does this prove about the composition of this part of the flame? (14) What is this part called?

By holding a platinum wire in various parts of the flame determine where the temperature is highest and indicate the

place on your drawing. Label your drawing to show the composition of the various parts of the flame and the names by which they are known.

(*e*) (1) Make a drawing of the Meker burner. (2) What is in the top of the tube of the burner? (3) What is its use? (4) Does a larger or smaller proportion of air enter the Meker burner or the Bunsen burner? (5) Why does the construction allow a different proportion? Light the burner. (6) What is the size of the flame compared with that of the Bunsen flame? (7) Which flame would you expect to be hotter? (8) Why? (9) What is the fact, as shown by testing with a platinum wire?

(*f*) Bring down upon the Bunsen flame a piece of fine copper gauze held in the tongs. (1) Where is the flame now? Hold a lighted match above the gauze. (2) What happened? (3) Was unburned gas passing through the gauze originally? (4) Why did it not burn? Turn off the gas and then turn it on again without lighting it. Hold the gauze about two inches above the top of the burner and apply a lighted match above the gauze. (5) Where is the flame? (6) What is below the gauze? Try some experiment to verify your answer. (7) What did you do, and what occurred? (8) Explain how the gauze acts to produce the results obtained in these experiments.

Light the gas with the air inlet of the burner open. (9) Why is air drawn into the burner?

Very slowly diminish the supply of gas until it takes fire at the base of the burner. (10) What is said to have occurred when this takes place? (11) Explain why it happened? (12) How does the appearance of the flame issuing from the burner differ from that of the luminous flame? After the gas has burned at the base of the burner for two or three minutes, turn it off, immediately turn it on again about half way and attempt to relight it in the ordinary manner. (13) Why did it become ignited at the base? (14) When a burner accidentally "strikes back," what should be done before attempting to relight the gas?

ACIDS, BASES, AND SALTS

EXPERIMENT 44. **Properties of Acids and Bases.**

Reference: **234–241.**

DISCUSSION: Solutions of a large number of substances fall into two very distinct classes, the members of each class having many identical properties but quite different properties from the members of the other class. The substances forming one class of solutions are termed acids and in all cases are compounds of hydrogen with an element the physical properties of which place it in the category of non-metals. Many compounds of this class contain oxygen also. The substances forming the other class of solutions are the hydroxides of the metals and are called bases. Among the bases, however, are included the hydroxides of all the metals, whether they form solutions or not. Those particular bases which are soluble are the alkalis. A solution of ammonium hydroxide, although not the hydroxide of a metal but the hydroxide of a radical, possesses the characteristic properties of solutions of the alkalis. A member of one class of solutions will react with a member of the other class to form water and a substance which belongs to a third class called salts. This type of reaction is termed neutralization.

MATERIALS: Dilute solution of acetic acid; of calcium, sodium, and potassium hydroxides; phenolphthalein, methyl orange, 10 per cent sodium hydroxide solution, woolen cloth, cotton cloth, silk, vinegar, sugar, litmus paper.

DIRECTIONS: (*a*) Put 5 c.c. of dilute hydrochloric, of dilute sulphuric, and of dilute acetic acids into each of three test tubes respectively, fill the test tubes with water, and shake them. Taste the solutions by putting a drop of each on the tip of your tongue with a glass rod. (1) Describe the taste of each. (2) In what experiments have you studied the action of acids upon metals? (3) What substance was formed in

every case? (4) In what experiments have you studied the action of acids upon carbonates? (5) What was formed? (6) Name three properties common to acid solutions. (7) What is the chemical composition of acids?

(*b*) Taste a solution of calcium hydroxide. Dilute solutions of sodium hydroxide, of potassium hydroxide, and of ammonium hydroxide as you diluted the acid solutions in (*a*) and taste them. (1) Describe the taste of each. (2) In what experiments have you studied the action of carbon dioxide on this class of compounds? (3) What was formed? (4) To what large class of substances do these compounds belong? (5) To what subdivision of this class do they also belong? (6) What is the chemical composition of bases?

(*c*) Add five drops of methyl orange to each of three test tubes one-third filled with water. Put five drops of sodium hydroxide solution into one test tube, of potassium hydroxide into the second, and of ammonium hydroxide into the third. (1) What colors did you obtain? (2) What is a test for an alkali?

Add dilute hydrochloric acid to one of the test tubes, dilute sulphuric to another, and dilute nitric to the third until a change of color occurs in each case. (3) What colors were produced? (4) What is a test for an acid?

Repeat these experiments with the substitution of five drops of phenolphthalein for the methyl orange. (5) What color is phenolphthalein in alkaline solution? (6) In acid solution? (7) What else have you used in testing for acids and alkalis? (8) Make a table consisting of the names of three indicators in the first column, their respective colors in acid solution in the second column, and their colors in alkaline solution in the third column.

(*d*) Measure out in your graduate 12 c.c. of 10 per cent sodium hydroxide solution, pour 10 c.c. of it into an evaporating dish and the remaining 2 c.c. into a test tube nearly full of water. To the solution in the evaporating dish add dilute hydrochloric acid little by little with constant stirring. Test the solution with litmus from time to time by touching the end of your stirring rod to a piece of litmus paper held in

your hand. Do not dip the paper into the solution. Do not lay the paper on the desk while making the test, as acid on the desk may contaminate the paper. Continue adding the acid until the solution in the dish gives an acid test. Now add some of the dilute solution of sodium hydroxide, which you have prepared in the test tube and which should be well shaken, until the contents of the dish is alkaline. Prepare a very dilute solution of hydrochloric acid by pouring 5 c.c. of the dilute acid into a test tube full of water. Pour this diluted solution into the dish little by little with constant stirring and frequent testing until the last small addition makes the contents of the dish acid.

Support the dish on a wire gauze and evaporate the solution to dryness. Toward the end of the evaporation when a solid begins to appear in the dish, the burner should be held in the hand and the flame waved under the dish, as otherwise the solid will spatter. When all the water has been driven off and the dish allowed to cool for several minutes, add 5 c.c. of water and again evaporate to dryness. (1) Describe the appearance of the substance left in the dish. Taste a little of it. (2) What familiar substance should you judge it to be? (3) Write the equation for the reaction by which it was formed. (4) Was the evaporation necessary to bring about this reaction or had it already taken place before the evaporation? (5) What is this kind of reaction termed? (6) What classes of substances enter into this kind of a reaction? (7) What substance is always one of the products? (8) To what class of substances does the other product belong?

(*e*) (1) What is the effect of vinegar upon litmus paper? (2) What kind of a substance does it contain?

To 10 c.c. of vinegar in a test tube add successive small amounts of sugar until it ceases to dissolve upon shaking and test the solution after each addition with litmus paper. (3) What was the result of the last test? (4) Is a sour taste still to be detected in the solution? (5) Did the sugar neutralize the acid? (6) What class of substances neutralizes acids? (7) Does sugar belong to this class? Verify your answer by an experiment. (8) What did you do and what was the result?

EXPERIMENT 45. **Quantitative Neutralization—Titration.**
Reference: **240.**

DISCUSSION: The object of this experiment is to find the concentration, namely the number of grams of acid per liter, of a solution of hydrochloric acid by determining how many cubic centimeters of the solution are required to neutralize a definite volume of sodium hydroxide solution, the concentration of which is known. For example if the sodium hydroxide solution was known to contain 20.00 grams of the compound per liter and if it were found that 10.00 c.c. of this solution was neutralized by 12.00 of hydrochloric acid solution, it is obvious that in the 10.00 c.c. of alkaline solution there was $20.00 \times \frac{10.00}{1000} = 0.200$ gram of sodium hydroxide and that in the 12.00 c.c. of acid solution there was present the weight of hydrochloric acid which would react with 0.200 gram of sodium hydroxide. This weight can be calculated by the familiar method of making a proportion between the molecular weights and the actual weights of the compounds. Having thus found the weight of hydrochloric acid in 12.00 c.c. of solution, the weight in 1000 c.c. is calculated by the aid of simple proportion.

In the actual experiment the relative volumes of alkali and acid required for neutralization are determined several times and the average of the results is taken as being more reliable than the value obtained from a single experiment.

Certain precautions must be taken to avoid errors in using a burette. (*a*) The burette must be washed clean, but enough water may adhere to the inside of it to dilute the solution. As drying the burette would be too tedious, it is rinsed out with some of the solution to be used. What adheres to the inside now is some of the solution itself, so no dilution occurs upon filling the burette.

(*b*) A bubble of air may be caught in the tip of the burette or in the rubber connector above it. If this bubble of air comes out during the neutralization, some solution from the burette will flow down to take its place and it will appear that this amount of solution has been used, whereas in reality only air

will have issued from the tip. All bubbles of air must therefore be removed from the burette after it has been filled and before it is used.

(*c*) In looking through the burette, the surface of the liquid appears as a curved line, which is called the meniscus. In reading the burette the position of the lowest part of the meniscus is taken as the height of the liquid. In observing its position on the scale on the burette, the eye must be on a level with the surface of the liquid. If the eye were too low the line of vision to the center of the meniscus would pass through a scale division on the burette below the true level and if the eye were too high the reverse would be true.

MATERIALS: Special solutions of sodium hydroxide and hydrochloric acid, phenolphthalein, two burettes.

DIRECTIONS: (*a*) Thoroughly wash out two burettes and clamp them to a ring stand in a strictly vertical position. Fill them with water and make sure that the tips do not leak. Remove the air bubbles in the tips by bending the tips upward and opening the pinch cocks slightly so that the water forces the air upward and out. (1) In actual use what error may be caused by allowing bubbles of air to remain in the tip? Practice the manipulation of the pinch cock, letting the water run out rapidly and then a single drop at a time. Read the height of the water in the burette. (2) What volume is indicated by the smallest division marks? With your eye five or six inches below the meniscus make a reading. Again make a reading with your eye an equal distance above the meniscus. (3) How great was the difference in readings? (4) Make a drawing to show the cause of this difference. (5) At what level should your eye always be in making a reading? (6) Record the proper reading. Run out 10 drops of water and (7) read again. (8) What is the volume of 10 drops? Determine again the volume of 10 drops, and (9) record all the readings. (10) What do you find to be the average volume of a drop? (11) To what fraction of a cubic centimeter, therefore, should you attempt to read a burette? (12) In making your record how many figures beyond the decimal point should appear? (13) If the reading chances to be a whole number

of cubic centimeters, should ciphers be placed after the decimal point? (14) Why?

(*b*) Let all the water run out of the burettes and after they have stood for three minutes run out the remaining drops which will have collected.

Procure in clean dry flasks 75 c.c. of the special solution of hydrochloric acid and of sodium hydroxide prepared for this experiment. Pour 5 c.c. of the acid into one burette and 5 c.c. of the alkali into the other. Take down in turn each burette, thoroughly rinse it with the 5 c.c. of solution by turning it up and down, replace it in position and run out the solution. (1) What is the object of this rinsing? Fill the burettes with their respective solutions, remove air from the tips and (2) record the initial reading of each. Run into a clean beaker about 15 c.c. of sodium hydroxide solution from the burette, add three drops of phenolphthalein and then slowly run in acid from the other burette with constant stirring until the indicator loses its color. Now add enough alkali to restore the color and again enough acid to destroy it. Continue adding small amounts of one and then of the other solution until one drop only will produce the change in the indicator. Finish by the addition of one drop of alkali to give a faint pink. Read the burettes, (3) record the readings, and subtract the initial readings from these to obtain the amounts of solution used. (4) Divide the number of cubic centimeters of acid by the number of cubic centimeters of alkali. (5) What does this quotient mean?

Repeat the experiment which you have just performed (6) recording the initial and final readings and making the same calculations as before. (7) Should you expect to get the same quotient as before? (8) Why? If your results differ from each other by more than 1 per cent continue making determinations until you can get two successive results which agree with each other to this accuracy. (9) What is the average of these accepted results?

One thousand cubic centimeters of the alkali solution contains 20.0 grams of sodium hydroxide. (10) 1 c.c. of this solution contains how many grams? (11) Calculate how many

grams of hydrochloric acid are required to react with this weight of sodium hydroxide. (12) In how many cubic centimeters of your solution is this weight of hydrochloric acid contained? (13) How do you know? (14) What weight of hydrochloric acid is contained in 1000 c.c. of your solution? (15) What was the object of the experiment? (16) What is the final answer?

EXPERIMENT 46. **Percentage of Acetic Acid in Vinegar. Relative Cost of Ammonium Hydroxide Solutions.**

Reference: **240.**

DISCUSSION: The object of this experiment is the determination of the percentage of acetic acid in vinegar. The experiment may be carried out as was Experiment 45 with the substitution of vinegar for hydrochloric acid, as described in the text. It is more usual in such cases, however, to measure out in a pipette a definite volume of the solution to be analyzed, to add to it an excess of a base, and to complete the neutralization with a standard solution of some acid. The latter method is employed in this experiment since it gives practice in additional manipulation and calculation.

MATERIALS: Standard solutions of hydrochloric acid and of sodium hydroxide, vinegar, phenolphthalein, methyl orange, "household ammonia," pipette, two burettes.

DIRECTIONS: (*a*) Set up and fill burettes with sodium hydroxide and hydrochloric acid solutions, as in Experiment 45 and (1) record the initial readings.

Rinse out a 10 c.c. pipette with a little vinegar, fill it with vinegar to the mark and let the vinegar run out into a clean beaker. Allow the pipette to drain for half a minute and blow out the drop, which will have collected in the tip, into the beaker. Add 25 c.c. of water and three drops of phenolphthalein. Run in slowly sodium hydroxide from the burette with constant stirring until the pink color of the indicator appears. Add enough hydrochloric acid from the other burette to destroy the color of the indicator and complete the neutralization as in Experiment 45. (2) Record the final readings of the burettes and calculate the volumes of each solution used.

(3) Referring to the ratio of alkali to acid determined in Experiment 45, calculate how many cubic centimeters of alkali is equivalent to 1.00 c.c. of acid. (4) How many cubic centimeters of alkali are equivalent to the volume of acid which you used in this experiment? (5) Subtract this volume from the total volume of alkali used. (6) Why is this subtraction made? (7) What does the remainder represent? Repeat the experiment, starting with another 10 c.c. of vinegar, until you obtain results which agree to within 5 per cent of one another. (8) What is the average of the accepted results? (9) Using this average and assuming that the sodium hydroxide solution contains 20.0 grams per liter, calculate the weight of the alkali used to neutralize the vinegar. (10) What weight of acetic acid ($HC_2H_3O_2$) will react with this alkali? (11) What was the percentage of acetic acid in the vinegar?

(*b*) Determine by a method similar to the one just used in (*a*) the relative concentrations of solutions of "household ammonia" and the ammonium hydroxide used as a laboratory reagent. Use methyl orange instead of phenolphthalein as an indicator. (1) Make records of your burette readings and such calculations as are necessary in order to state the average volumes of acid required to neutralize 100 c.c. of each solution of ammonium hydroxide. Inquire from the instructor the cost per liter of each of the ammonium hydroxide solutions. (2) Which is cheaper for equivalent amounts of ammonia? (3) In what ratio?

SOLUTIONS

EXPERIMENT 47. **Formation of Ions.**

Reference: **243.**

DISCUSSION: When two samples of material have the same characteristic properties, the fact is summarized by saying that they are the same substance. Likewise if two mixtures exhibit a number of characteristic properties in common, it is concluded that they each contain some of the same substance. Such mixtures, with similar properties, are common in certain classes of solutions, as for example in solutions of acids, where similarity in taste, action with indicators, with metals, etc., have been noted. It appears, then, that all acid solutions are mixtures containing some of the same substance, but since these solutions were made from different acids, different substances, they must have reacted to form some of the same new substance in every case. By no type of reaction previously studied could the facts be so well explained as by the assumption that every acid in solution breaks up into two electrically charged substances one of which is always hydrogen and that this is the substance which gives the similar properties to all acid solutions. This particular kind of reaction which produces electrically charged substances in solution is termed electrolytic dissociation and the charged substances are called ions.

In the same way the common properties of all alkaline solutions are accounted for by the formation of hydroxyl ions, of all solutions of chlorides by the formation of chloride ions, of all sulphates by the formation of sulphate ions, etc. A solution, thus, is a mixture of two or more ionic substances together with the original substance put into the solution, provided the reaction of dissociation has not been complete. It does not follow, however, that all substances react this way when dissolved, or that all compounds of hydrogen form hydrogen ions, of chlorine, chloride ions, etc.

The reaction of dissociation is caused by the presence of the water; but when the water is again removed from a solution, the reaction tends to become reversed and when finally all of the water has been taken away, only the original substance remains.

MATERIALS: Litmus paper, glacial acetic acid, aluminium, solutions of sodium chloride, potassium chloride, magnesium chloride, silver nitrate, and silver sulphate; potassium chlorate, sugar, alcohol, chloroform.

DIRECTIONS: (*a*) (**1**) What was the action on litmus paper of the solutions of acids studied in Experiment 44? Fill a *perfectly dry* test tube one-quarter full of glacial acetic acid (this is a pure substance and contains no water) and transfer a drop of it with a rod to a piece of blue litmus paper. (**2**) How does the result differ from that obtained with the acid solutions in Experiment 44? Add a drop of water to the acid on the litmus paper. (**3**) What happened? (**4**) What property is exhibited by a solution of acetic acid which is not possessed by the pure substance itself? (**5**) When acetic acid is dissolved in water is any new substance formed? (**6**) What is the evidence? (**7**) Do other acids when dissolved in water form the same substance? (**8**) What is the evidence? (**9**) What is the name of the substance? (**10**) What is its chemical composition? (**11**) How is it essentially different from other substances previously studied? (**12**) Can it be obtained in a pure state out of solution? (**13**) What happens to it when a solution containing it is evaporated? (**14**) What is a test for it?

Drop a small piece of aluminium into the glacial acetic acid in the test tube and warm it. (**15**) What happened? Add an equal volume of water and again warm it. (**16**) What difference in result is occasioned by the presence of the water? (**17**) Explain how this experiment substantiates the conclusion reached from the one with the litmus paper.

(*b*) To 5 c.c. each of solutions of sodium chloride, of potassium chloride, and of magnesium chloride in separate test tubes add a few drops of silver nitrate solution. (**1**) What was formed in each case? (**2**) What common property was

possessed by the three original solutions? (3) What substance in solution do they possess in common?

Repeat the experiment with the substitution of silver sulphate solution for silver nitrate solution. (4) Record the results of the experiments. (5) What substance besides water exists in both silver nitrate and silver sulphate solutions? (6) Write equations in the ionic form for the reaction which took place in all six experiments. Cross out in each equation the formulas which occur on both sides of the equation. (7) What remains in each case? (8) Did the same reaction or six different ones take place?

Dissolve a few crystals of potassium chlorate and add some silver nitrate to the solution. (9) What occurred? (10) Do all salts containing chlorine form chloride ions? (11) What is the evidence?

(*c*) (1) Referring to Experiment 44, what is the evidence that all alkaline solutions contain a common substance? (2) What is its name? (3) What is a test for it?

(*d*) Test for the presence of the hydrogen ion in a solution of sugar ($C_{12}H_{22}O_{11}$); of hydroxide ion in alcohol (C_2H_5OH); and of chloride ion in chloroform ($CHCl_3$). (1) (2) (3) State what you did, what you observed, and what you concluded in each case.

EXPERIMENT 48. **Electrical Conductivity of Solutions.**

Reference: **242–244.**

DISCUSSION: Whether or not a given liquid is a good conductor of electricity can be determined roughly by attempting to pass through the liquid a current which has a lamp included in the circuit. If the lamp glows a current is passing and the liquid is proved to be a good conductor; if the lamp does not glow the liquid is not a good conductor.

In this experiment the conductivity of some pure liquids is tried, then that of some solutions. It will be found that all solutions do not behave alike in this respect, only those of certain classes of substances being good conductors. Upon this fact and certain other peculiarities of these same substances, which were studied in Experiment 47, the conception of electrolytic dissociation is based.

MATERIALS: Glacial acetic acid, distilled water, alcohol, solutions of acetic, of sulphuric, and of hydrochloric acids, of sodium and potassium hydroxides, of sodium chloride, of potassium nitrate, and of sugar, conductivity apparatus.

DIRECTIONS: The apparatus consists of two carbon rods about 5 mm. in diameter and 10 cm. long held in one-hole stoppers, which are fitted into holes 2 cm. apart in a thin piece of wood 15 cm. long and 5 cm. wide. The upper ends of the rods are connected by binding posts with lead wires which are attached to terminals of a board carrying a socket and lamp which in turn is connected through a switch with a lighting circuit or other source of current. The piece of wood which carries the carbon rods is held in the clamp of a ring stand, whereby the rods may be lowered into a beaker containing the liquid to be tested.

CAUTION: *Be sure that the switch is left open, that is, so no current can pass through it, except when actually making the tests.*

Select the smallest beaker into which the rods of the apparatus can be lowered. Wash off the rods and wipe them dry (Handle them carefully as they are fragile). Fill the beaker one-third full of glacial acetic acid, (this is a pure liquid which contains no water) lower the rods into the acid, turn on the switch and observe whether or not the lamp glows. *Turn off the switch.* (**1**) Record the result of this and the following experiments by making a column of the names of the substances tested and noting at the right of each the behavior of the lamp and the conclusion as to the conductivity of the liquid. Pour the acid into the bottle marked "Acetic Acid Returned," wash the rods and the beaker, and then determine the conductivity of distilled water in the same way that you tried that of glacial acetic acid. Try the conductivity of alcohol and of the following solutions in turn: acetic acid, dilute sulphuric acid, dilute hydrochloric acid, sodium hydroxide, potassium hydroxide, sodium chloride, potassium nitrate, alcohol, sugar. Ten cubic centimeters of each solution should be put into the beaker and enough water added to fill the beaker a third full. The carbon rods must be carefully rinsed

after each trial. (1) Record the results of the experiments. (2) Are pure liquids conductors? (3) Are all solutions conductors? (4) What classes of substances form conductive solutions? (5) Is anything new formed when an acid, like acetic in this experiment, is put into water? (6) What is the evidence? (7) What is this kind of reaction called? (8) What is the general name of the substance formed? (9) What classes of substances act thus? (10) Does the reaction take place when the solution is made, or when the current passes? (11) What carries the current through the solution?

EXPERIMENT 49. **The Determination of the Heat of Neutralization.**

Reference: **245.**

DISCUSSION: Neutralization is the interaction of an acid and a base, as the result of which a salt and water are formed. From the standpoint of the theory of elctrolytic dissociation, in neutralization the hydrogen ions furnished by the acid unite with the hydroxyl ions furnished by the base. If the solutions are sufficiently dilute, both the acid and the base are completely dissociated, and the only change that occurs when they are mixed is the formation of undissociated water from its ions. It follows that equal volumes of equivalent solutions of acids and of bases should produce the same amount of heat when neutralization takes place. The experiment described below is designed to test this conclusion. Equal quantities of normal solutions of several acids and bases are mixed and the heat developed in each case is measured. This is done by determining the rise in temperature that occurs when the solutions are mixed. Since dilute solutions are used, it is assumed in the calculations that the specific heat of the resulting salt solutions is equal to that of water, which is 1. If the volume of the solution is multiplied by the rise in temperature, the product is the number of calories set free.

A part of the heat, however, will be used in raising the temperature of the surrounding air, the thermometer, and the vessel containing the solution. As a consequence, a correction must be made, as was done in Experiment 31.

MATERIALS: Normal solutions of potassium hydroxide, sodium hydroxide, hydrochloric acid, and sulphuric acid.

(If the solutions are not furnished, they can be prepared by dissolving separately in 100 c.c. of water in each case 8.0 grams of sodium hydroxide, 11.2 grams of potassium hydroxide, 5.6 c.c. of concentrated sulphuric acid, and 16 c.c. of concentrated hydrochloric acid, and then adding water to make the volume of each solution 200 c.c. The solutions should be cooled to room temperature before being used.)

DIRECTIONS: (*a*) Measure 50 c.c. of a normal solution of sodium hydroxide in a graduated cylinder and pour it into a 200 c.c. beaker. Place a thermometer in the solution and (**1**) record the temperature to a tenth of a degree. Measure 50 c.c. of the solution of sulphuric acid in the graduate and pour it into the beaker, and at once observe the thermometer. Stir and record to tenths of a degree the highest point reached. The temperature changes rapidly and the reading must be made promptly. Repeat the neutralization in the same way to obtain a second observation and average the results.

(*b*) To determine the correction that must be added to the observed rise in the thermometer pour out the solution used in (*a*) above, wash the beaker with water at room temperature, and add to it 50 c.c. of water at this temperature. Insert the thermometer and (**1**) record the temperature to tenths of a degree. Next heat in a small flask 50 c.c. of water to a temperature about twice as many degrees above the temperature of the cold water as the rise in temperature observed in (*a*) above. (The warm water will be from 12 to 14 degrees above room temperature.) Read the temperature of the warm water to tenths of a degree, place the thermometer in the beaker and at once pour the warm water into it and read the temperature of the mixture. (**2**) Calculate the temperature half-way between the two extremes used and subtract from it the temperature observed when the two portions of water were mixed. Repeat this part of the experiment and add the average of the results to the average temperature determined in (*a*).

(**3**) Calculate the number of calories set free in the reaction

(rise in temperature × volume) and from this value the number set free when 1 gram-molecular-weight of the base is used.

(*c*) Determine in the way described in (*a*) above the rise in temperature when 50 c.c. of the hydrochloric acid solution is mixed with 50 c.c. of sodium hydroxide. (**1**) Calculate the molecular heat of neutralization.

(*d*) Repeat using 50 c.c. of potassium hydroxide and 50 c.c. of sulphuric acid. (**1**) Calculate the results as before.

(*e*) Repeat using 50 c.c. of potassium hydroxide and 50 c.c. of hydrochloric acid. (**1**) Calculate the results as before, and (**2**) obtain the average of all the experiments. (**3**) By what percentage does your result differ from the true value which is 13,600 calories?

(**4**) Does the same reaction, or do different reactions take place between the different acids and bases? (**5**) How do the results of this experiment confirm your answer? (**6**) What is the reaction involved?

EXPERIMENT 50. **Electromotive Series.**

Reference: **248–252.**

DISCUSSION: All metals tend to pass from the condition of the free element to that of ions in solution; they differ markedly in the strength of this tendency, which is called the solution pressure of the element. In order to pass into the ionic condition, however, a metal must acquire positive electrical charges. These charges can be gained from the ions already in a solution of a salt into which the metal is put, provided the solution tension of the metal is greater than the solution tension of the metal which would be formed from the existing ions by the loss of their charges. In other words if the metals are arranged in the order of the magnitude of their solution pressures, any metal may form ions at the expense of any metal below it in the series and existing as ions in solution. Or conversely, by observing which metals will replace which others from solutions, the order of their relative solution tensions may be determined. Since hydrogen as well as the metals forms positive ions and may be replaced by certain metals, it too has a place in the series.

Not only do the metals tend to form positive ions, but likewise the non-metals tend to form negative ions and may be arranged in a series in accordance with the value of this tendency. These series are known as the electromotive series.

MATERIALS: Zinc, copper, solutions of copper sulphate, sodium chloride, mercuric chloride, potassium bromide, potassium iodide, iodine, bromine water, chlorine water, carbon disulphide.

DIRECTIONS: (*a*) Slip strips of clean zinc into test tubes containing solutions of copper sulphate and of sodium chloride. (1) What happened in each case? Slip a strip of clean copper, or a piece of clean heavy copper wire, into a solution of mercuric chloride. (2) What occurred? (3) Arrange zinc, sodium, copper, and mercury in the order of their tendency to form ions. (4) What is this tendency termed?

Try the action of dilute hydrochloric acid on zinc, and on copper. (5) What happened? Insert hydrogen in its proper place in the list of metals in (3). (6) What is the name of the series of which this list is a part? (7) Would you expect hydrochloric acid to act upon mercury? (8) Why?

(*b*) As a preliminary to this experiment make yourself familiar with the color of iodine in a solution of carbon disulphide, and of bromine in the same solvent. Since both of these elements are more soluble in carbon disulphide than in water, the former will extract them from water solutions. PRECAUTION: *Do not use carbon disulphide near a flame, since it is inflammable.* To 10 c.c. of water in a test tube add 1 c.c. of bromine water, 2 c.c. of carbon disulphide and shake the test tube. (1) What color is bromine dissolved in carbon disulphide? Repeat the experiment substituting a solution of potassium bromide for the bromine water. (2) What is the appearance of the carbon disulphide? (3) Will the bromide ion color carbon disulphide? (4) What does give it a color? Repeat the experiment using 1 c.c. of iodine solution instead of bromine water. (5) What color is iodine dissolved in carbon disulphide?

Having now become familiar with the colors of bromine and of iodine in carbon disulphide, add 10 c.c. of water, 1 c.c. of chlorine water and 2 c.c. of carbon disulphide to 2 c.c. of

potassium bromide in one test tube and to 2 c.c. of potassium iodide in another test tube and shake them. (6) What was formed in each case respectively? Repeat the experiment with the potassium iodide solution substituting for the chlorine water bromine water, but adding it only a few drops at a time and shaking after each addition. (7) What was formed? (8) Arrange bromine, chlorine, and iodine in the order of their solution pressures.

Experiment 51. **The Determination of Molecular Weights from the Freezing Points of Solutions.**

Reference: **254** (Read also **253**).

Discussion: The freezing point of a solution is always lower than that of the pure solvent. It has been shown as the results of many experiments that in the case of a substance which is not ionized (a non-electrolyte) the lowering of the freezing point is proportional to the concentration of the solution. The concentration is usually expressed as the number of grams of the dissolved substance contained either in 1 liter of the solution or in 1000 grams of the solvent. The freezing point of a solution made by dissolving 1 gram-molecular-weight of any non-electrolyte in 1000 grams of water is —1.86° C. This fact makes it possible to determine the molecular weight of non-electrolytes which are soluble in water. The experiment described below shows how this can be done.

The freezing point of a solution can be determined in two ways. Either the solution can be cooled until the temperature is reached at which ice begins to form, or ice can be added to the solution and the temperature noted at which the thermometer after falling, begins to rise. In the latter method the temperature observed is not the freezing point of the solution originally used, for as the temperature falls, some ice melts and the solution becomes diluted. The observed temperature is the freezing point of the solution present when the thermometer is read. In order to determine the concentration of the solution, the ice is separated from it by pouring the mixture through a funnel into a graduated cylinder, as soon as the reading of the thermometer has been made. The volume of the

solution is noted and from this and the weight of the dissolved substance used, the concentration of the solution which had the observed freezing point can be calculated.

This last method is used in the experiment described below, because it is more readily carried out than the apparently simpler method of determining directly the freezing point of a solution of known concentration.

The data needed for the calculation of the molecular weight are the weight of substance used, the volume of the solution when the temperature was read, and the freezing point observed.

MATERIALS: Sugar, ice, large funnel.

DIRECTIONS: (*a*) Before making the experiment the thermometer to be used must be tested to see if it registers correctly at the freezing point of water. Fill a 200 c.c. beaker full of ice, cracked in pieces approximately the size of a walnut. Wash the ice by adding water until it is covered; stir and pour off the water. Next just cover the ice with water and insert the thermometer so that the zero point is just above the surface of the liquid. Stir slowly and when the temperature is constant read the thermometer. When making the reading have your eye at the same level as the top of the column of mercury. Estimate the position of the mercury to tenths of a degree. (1) Record the freezing point and use this value in calculating later the lowering of the temperature in the following experiment. Pour off the water and keep the ice for the next part of the experiment.

(*b*) Weigh to centigrams on a filter paper approximately 30 grams of sugar. Pour the sugar into a beaker, add 50 c.c. of water, and stir until the sugar has dissolved. Add next enough cracked ice so that some of the pieces rest on the bottom of the beaker. The pieces should be of such a size that after melting somewhat they will not pass through the funnel to be used later. Stir slowly with the thermometer. At first the temperature will fall and then it will slowly rise. At this point read the thermometer to the nearest degree and estimate to tenths of a degree. In reading the thermometer be sure that your eye is at the same level as the top of the column

of mercury. Immediately pour the contents of the beaker into a funnel, large enough to hold all the ice, supported in a 100 c.c. graduated cylinder. (If only a 50 c.c. graduated cylinder is available, the funnel may be supported in a flask, and the volume of the solution subsequently measured.) Remove the funnel, and read the volume of the solution.

(1) How many grams of sugar were in your solution? (2) Since the depression of the freezing point is proportional to the concentration, how many grams would be necessary to make the solution freeze at —1.86° C. (3) How many grams of sugar would be contained in 1000 c.c. of such a solution? (4) Why is this number the molecular weight of sugar? (5) Calculate the theoretical molecular weight of sugar from its formula which is $C_{12}H_{22}O_{11}$. (6) What was the percentage error in your determination?

EXPERIMENT 52. **The Determination of the Degree of Dissociation of Electrolytes from the Freezing Point of Solutions.**

Reference: **253–254.**

DISCUSSION: When an electrolyte dissolves in water the solution contains, in addition to the undissociated compound, ions, the number of which depends upon the concentration of the solution. The number of particles present which affect the freezing point of the solution is, consequently, greater than in the case of a non-electrolyte. If a salt, sodium chloride for example, yields two ions when it dissociates, its effect upon the freezing point of water will be twice that of a non-electrolyte at the same concentration, provided it is completely dissociated; if it is but partly dissociated the effect will be less. From this effect it is possible to calculate by the following method the extent of dissociation of the salt.

If we consider 100 molecules of sodium chloride, and let a represent the number of these molecules which are ionized, then evidently a is the per cent ionized and $100 - a$ is the per cent not ionized. Since sodium chloride yields two ions, the number of ions is $2a$. The total number of particles in the solution is the sum of the un-ionized molecules and the number of ions, which in this case is $(100 - a) + 2a$ or $100 + a$. The lowering

of the freezing points of two solutions having the same volume are proportional to the number of particles present in the solutions. Consequently,

$$\frac{100}{100 + a} = \frac{\textit{lowering of freezing point if no ionization}}{\textit{lowering of freezing point observed}}$$

To calculate the extent of dissociation it is only necessary to substitute in the formula above the values of the expressions written out in words and solve for *a*.

If a solution of sodium chloride is studied, the lowering of the freezing point observed is the quantity experimentally determined. The lowering of the freezing point if no ionization occurred is the value calculated from the freezing point of a solution of a non-electrolyte of the same molar concentration as that used in the experiment. From the weight of the salt used and from the volume of the solution the molar concentration is known.

MATERIALS: Pure sodium chloride, ice, large funnel.

DIRECTIONS: Determine the reading of your thermometer in pure ice and water. Weigh to centigrams on a piece of filter paper approximately 15 grams of sodium chloride. Pour the salt into a 200 c.c. beaker and dissolve it in 50 c.c. of water. Add cracked ice until some of it rests on the bottom of the beaker and stir slowly with a thermometer. At first the temperature of the mixture will fall; when it begins to rise (1) record the temperature to tenths of a degree and immediately pour the mixture into a large funnel supported in a 100 c.c. graduated cylinder. Remove the funnel, and (2) record the volume of the solution.

(3) How many grams of salt did your solution contain and in what volume? (4) What weight of salt would be contained in 1000 c.c. of a solution of this concentration? (5) What is the molecular weight of sodium chloride? (6) How many times molar was the concentration of your solution? (7) What is the freezing point of a molar solution? (8) What would have been the freezing point of your solution had no dissocia-

tion occurred? (9) What was the observed freezing point? (10) Substitute these last two values in the equation given in the discussion and solve for *a*. (11) What does *a* represent? (12) What is the final result of this experiment?

CHEMICAL EQUILIBRIUM

EXPERIMENT 53. **Reversible Reactions.**

Reference: **260–262.**

DISCUSSION: If two substances, *A* and *B*, react and form the substances *C* and *D*, and if the latter can interact to form the former, the reaction involved is said to be reversible. This fact is represented by writing the equation for the reaction in a general form as follows:

$$A + B \rightleftharpoons C + D$$

If any one of the substances involved in the reaction is constantly removed as the reaction proceeds, the reaction runs to completion in one direction. In the general case given above if *A* or *B*, for example, is removed all of *C* and *D* are transformed into *A* and *B*; likewise if *C* or *D* is removed *A* and *B* are completely converted to *C* and *D*.

The experiment described below is designed to illustrate the effect of the removal of one of the products of a reversible reaction. The reaction studied is that represented by the following equation:

$$NaCl + H_2SO_4 \rightleftharpoons NaHSO_4 + HCl$$

To force the reaction in the direction indicated from left to right in the equation, dry sodium chloride is treated with concentrated sulphuric acid. Under these conditions hydrogen chloride, which is a gas, escapes and all the salt is changed to sodium hydrogen sulphate. To force the reaction in the opposite direction an aqueous solution of sodium hydrogen sulphate is treated with concentrated hydrochloric acid. Since sodium chloride is only sparingly soluble in strong hydrochloric

acid, it is formed and separates out as a precipitate. As a result, the sulphate is changed to chloride.

MATERIALS: Sodium chloride, concentrated hydrochloric acid, solution of ferric chloride, ammonium chloride, ammonium thiocyanate.

DIRECTIONS: (*a*) (*Hood*) To 10 grams of dry sodium chloride contained in a small beaker add 13 c.c. of concentrated sulphuric acid. In 2 or 3 minutes place the beaker on a wire gauze, cover it with a watch glass, and heat it with a small flame that just touches the gauze. Blow across the top of the beaker. (1) What happened? (2) What is thus shown to be escaping from the beaker?

When all the salt has dissolved and the beaker contains a clear liquid remove the flame, let the substance cool until it crystallizes, and observe the appearance of the crystals. Next add 20 c.c. of water and heat cautiously until the salt has dissolved. Cool the beaker to room temperature, and after pouring about half of the solution into a test tube, for future use in (*b*), place the beaker in cold water. When crystals have formed in the beaker, filter them off and (3) describe their form. Scrape the crystals into a watch glass and add to them a few drops of concentrated sulphuric acid. (4) Was any hydrochloric acid given off? (5) How do you know? (6) Were the crystals sodium chloride? (7) What is the evidence? (8) What then were the crystals? (9) Write an equation for the original reaction. (10) Why did it take place?

(*b*) Add to the solution in the test tube saved from (*a*) an equal volume of concentrated hydrochloric acid. (1) What happened? Filter off the precipitate and press out as much of the liquid as you can with a spatula. Remove the filter paper, open it out flat, and press it and the salt between filter papers. Remove the salt, place it on a dry piece of paper, and set it aside. Dissolve a pinch of the salt in a few drops of water on a watch glass and set it aside. (2) Describe the appearance of the crystals formed when the water has evaporated.

Using the main portion of the solid on the filter paper, determine whether or not it is sodium chloride. (3) What

did you do? (4) What was the result? (5) What do you conclude? (6) Write an equation for the formation of the precipitate? (7) What is the relation of this reaction to that in (*a*)? (8) Why did this reaction take place? (9) Make a general statement as to the factors which cause a change in the equilibrium in a reversible reaction.

(*c*) To 20 c.c. of water in a test tube add exactly from a glass tube 5 drops each of solutions of ferric chloride and of ammonium thiocyanate. Pour a quarter of the solution into each of three other test tubes and to one add 2 c.c. of ferric chloride, to another 2 c.c. of ammonium thiocyanate and to a third 5 c.c. of ammonium chloride solution. Compare the colors of these three solutions with that of the fourth which was kept as a standard of reference. (1) How did each of the three solutions change in color? (2) Write an equation for the equilibrium existing upon adding ferric chloride to ammonium thiocyanate. (3) What is the colored compound? (4) Explain the causes of the changes in color upon adding the ferric chloride, the thiocyanate, and the ammonium chloride respectively.

EXPERIMENT 54. **The Determination of the Effect of Increase in Temperature on the Rate of a Reaction.**

Reference: **265.**

DISCUSSION: The rate at which most reactions take place is approximately doubled for an increase in temperature of ten degrees. In the experiment described below determinations at two temperatures are made of the time required to reduce a solution of potassium permanganate by means of iron and sulphuric acid. Potassium permanganate has a purple color and is converted by reduction into a colorless compound. All the conditions except the temperature are kept constant by using equal volumes of the same solution in test tubes of the same size; the iron is used in the form of nails so that the surface of the metal in each case is the same.

The results are to be plotted on Fig. 11, expressing time on the vertical lines (abscissae) and the temperature on the horizontal lines (ordinates). The theoretical

curve for the relation of time to temperature is also drawn, as based on the assumption that the speed of the reaction is doubled for an increase of temperature of ten degrees. The observed values may then easily be compared with the theoretical.

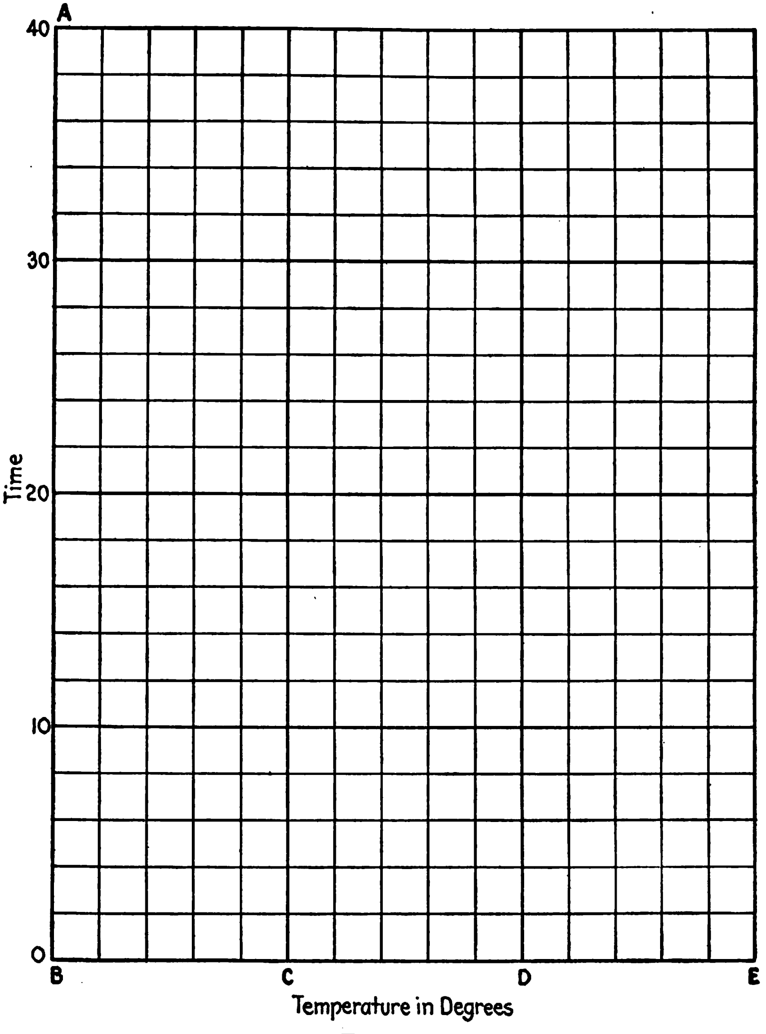

FIG. 11.

MATERIALS: A solution is furnished which is made by dissolving 1 gram potassium permanganate in 1 liter of water and adding 200 c.c. concentrated sulphuric acid. Two wire nails $2\frac{1}{4}$ inches long, watch with second hand.

DIRECTIONS: (*a*) Measure accurately in a graduate 10 c.c. of the solution of sulphuric acid and potassium permanganate and pour it into a dry test tube. Place this tube in a large beaker filled with water at room temperature. (**1**) Note the temperature of the water to tenths of a degree, which should be the same as that of the solution in the test tube. (**2**) Note the time to seconds as a nail is carefully dropped into the test tube.

While the reaction is taking place, measure accurately 10 c.c. of the acid solution into a dry test tube and place the latter in a beaker full of water. Heat the water until its temperature is about 25 degrees above room temperature, remove the beaker from the source of heat, (**3**) note to tenths of a degree the temperature of the acid solution, and (**4**) record the time when a nail is placed in the test tube.

(**5**) Record the time when each of the acid solutions loses its color. (**6**) Note the temperature of the warm solution when its color disappears and use the mean of this temperature and that observed at the beginning of the experiment as the temperature of this solution.

(*b*) To prepare the theoretical curve for the relation of speed of the reaction to time, proceed as follows using the diagram on Fig. 11. (**1**) Label the point *B* the temperature of the first tube (room temperature), and label the points *C, D,* and *E,* which are respectively 10, 20, and 30 degrees above room temperature. Make a cross on the line rising from *B* (the line *BA*) to indicate the time of the reaction at room temperature. In choosing the number of divisions which you will let represent one minute, select such a number as will bring the cross near the upper end of the line. On the vertical line rising from *C* make a cross half as high above the base line as the cross on the line *BA,* since at temperature *C* the reaction will require only half as much time as before. On the line rising from *D* make a cross at half the height of that above *C,*

and on the line rising from *E* a cross at half the height of that above *D*. Draw a smooth curve through the crosses. (2) What does this curve represent?

Mark with the letter *F* the position on the base line corresponding to the higher temperature at which you carried out the reaction. On the line rising from *F* make a cross at the height corresponding to the time which elapsed in the experiment. (3) If the speed of this reaction had increased exactly at the rate of doubling for every ten degrees rise, where would this cross above *F* have fallen in relation to the curve?

SULPHUR AND SULPHIDES

EXPERIMENT 55. **Allotropic Forms of Sulphur.**

Reference: **273.**

DISCUSSION: Sulphur can exist in two different crystalline forms and two different liquid forms, one of which when suddenly cooled becomes so stiff that it closely resembles a solid. The variety of sulphur which is rhombic in crystalline form is stable up to 96° and the other variety, the monoclinic, from this temperature to its melting temperature 119°. The rhombic sulphur can be heated temporarily above 96°, and the monoclinic sulphur can be cooled below this temperature, because the rearrangement of the material of a solid takes place slowly; eventually, however, all sulphur below 96°, the "transition temperature," becomes rhombic and above this temperature monoclinic.

In preparing crystals of any substance, one of two methods is used; either the substance is melted and then slowly cooled, when crystals are formed, as in the case of ice, or the substance is dissolved and then the solvent evaporated, when the material may come out of solution as crystals, as in the case of crystallizing sugar from water solution. In the case of rhombic sulphur the first method can not be used, because crystallization must take place below the transition temperature, which in turn is below the solidification temperature of liquid sulphur. The second method must therefore be resorted to and evaporation carried on below 96°. For preparing monoclinic crystals the first method is suitable, since this form is stable directly below the solidification temperature of the liquid.

All liquids become more viscous upon cooling. If some liquids are suddenly cooled below their solidification temperature they become so stiff and rigid that they appear solid and may remain in this condition for a long time, since the

material does not easily arrange itself in a crystalline or true solid form because of the great viscosity. Glass is such a supercooled liquid. The dark colored liquid sulphur, when suddenly cooled, affords another example of this phenomenon and is thus often referred to as a third solid amorphous form.

MATERIALS: Roll sulphur, flowers of sulphur, carbon disulphide.

DIRECTIONS: (*a*) Heat a test tube one-third full of sulphur slowly until it boils, observing changes in color and viscosity during the process, and then pour the boiling sulphur into an evaporating dish filled with cold water (1) What was the color of the sulphur just as it melted? (2) What was the color upon further heating? (3) What changes in viscosity occurred? (4) How many varieties of liquid sulphur exist?

Examine the sulphur which was poured into the water. (5) What is its most striking physical property? (6) What is this form called? Preserve it until the next exercise and then answer the following questions. (7) How has it changed in appearance? (8) As what form does it now chiefly exist? (9) Why did it not assume this form immediately?

(*b*) Fill a test tube nearly full of roll sulphur and melt it very slowly at as low a temperature as possible, not allowing it to become dark. Pour the melted sulphur into a filter paper folded as for filtering, but held by its edge in your hand instead of in a funnel as usual. As soon as crystals begin to form in the filter paper, pour the remaining melted sulphur quickly into a dish of cold water. Unfold the filter paper and examine the crystals. (1) Record the properties of the crystals, especially their shape, color, luster, and brittleness. (2) What is this form of sulphur termed? (3) Why is it so named? (4) Between what temperatures is it stable? (5) At about what temperature is it now? (6) How do you account for its existence at this temperature? Preserve it until the next exercise and then answer the following questions. (7) How has it changed in appearance? (8) As what form does it now exist? (9) Why did it change?

(*c*) To 3 grams of flowers of sulphur (powder) in a test tube add about 5 c.c. of carbon disulphide. (Remember the

precaution to be observed in using this compound, Experiment 50). Shake the tube until all the sulphur is dissolved, then pour the clear solution into an evaporating dish to crystallize in the hood where there is no flame. Cover the dish with a piece of filter paper to retard evaporation. Watch the crystallization toward the end, and, if perfect crystals form, remove them with the forceps. Allow the liquid to evaporate almost entirely, then remove and dry the crystals. (**1**) Examine them as in (*b*) and record their properties. (**2**) What is the name of this kind of sulphur? (**3**) Can it be formed directly from melted sulphur? (**4**) Why? (**5**) Name all the different forms of sulphur. (**6**) Which is the stable form at room temperature?

EXPERIMENT 56. **Preparation of Hydrogen Sulphide.**

Reference: **277.**

DISCUSSION: As was pointed out in Experiment 53, hydrochloric acid, or as it may be called hydrogen chloride, was formed by the double decomposition between a chloride and an acid, under such conditions that the hydrogen chloride could escape. In a similar way hydrogen sulphide may be prepared by double decomposition between a sulphide and an acid. Since hydrogen sulphide is only slightly soluble in water, the reaction may take place in dilute solution, so that a solution of hydrochloric acid may be used as the necessary acid. The sulphides of many metals are so insoluble that this double decomposition takes place chiefly in the reverse direction and so these sulphides can not be used for the preparation of hydrogen sulphide.

MATERIALS: Iron sulphide, lead acetate.

DIRECTIONS: (*All experiments with hydrogen sulphide gas should be performed in the hood.*)

Set up an apparatus similar to the one used for the preparation of chlorine in Experiment 21. Put 15 grams of iron sulphide in the test tube and pour in enough dilute hydrochloric acid to seal the end of the thistle tube. Gas should begin to be evolved at once and when the evolution slackens a little concentrated hydrochloric acid may be added, a few cubic

centimeters at a time. Collect the gas, which is heavier than air, in dry bottles and determine when each bottle is full by testing with a piece of filter paper which has been dipped into a solution of lead acetate. Hydrogen sulphide turns lead compounds black. When two bottles of gas have been collected, allow the hydrogen sulphide to bubble into half a bottleful of water for ten minutes and then empty the contents of the generator into the bottle in the hood provided for the purpose. (1) Write the equation for the reaction by which hydrogen sulphide was prepared. (2) To what class of reactions does it belong? (3) Why does it take place to completion? (4) Why do not all sulphides act like iron sulphide? (5) Write the equation for the reaction taking place in testing for hydrogen sulphide.

EXPERIMENT 57. **Properties of Hydrogen Sulphide.**

Reference: **280–283.**

DISCUSSION: In the burning of hydrogen sulphide appear analogies between the hydrogen compounds of sulphur and of carbon in that both form water and the dioxide of the nonmetal, and that both are unstable at the temperature of the flame, and deposit the uncombined element when burning in an insufficient supply of oxygen.

In solution the acidic properties of hydrogen sulphide appear and under the name of hydrosulphuric acid the compound may be compared with hydrochloric acid.

MATERIALS: Splinter, concentrated nitric acid, litmus paper, lead acetate, copper sulphide, zinc.

DIRECTIONS: (*a*) Thrust a burning splinter into a bottle of hydrogen sulphide and at once invert a dry bottle of air over it. (1) What happened? (2) What was deposited on the sides of the bottles? (3) Is hydrogen sulphide stable at the temperature of the flame? (4) What is the evidence? (5) Write the equation for the complete oxidation of hydrogen sulphide.

(*b*) Into another bottle of the gas pour a few drops of concentrated nitric acid, cover the bottle and shake it gently. (1) What insoluble substance is formed? (2) By what kind

of reaction was it formed? (3) What kind of an agent is nitric acid? (4) What kind of a reaction did the nitric acid undergo? (5) What kind of an agent is hydrogen sulphide?

(*c*) (1) Is hydrogen sulphide soluble? (2) What is the evidence? Examine the bottle on the table which was filled with a solution of hydrogen sulphide but has been exposed for some days to sunlight and air. (3) What insoluble substance is in the bottle? (4) Does the solution smell of hydrogen sulphide? (5) Is hydrogen sulphide stable in solution?

(*d*) Test the solution of hydrogen sulphide, which you prepared in Experiment 56, with blue litmus paper. (1) What does the test show? (2) Why is the action less marked than it would be in a solution of hydrochloric acid of equal concentration? (3) Is hydrogen sulphide a weak or a strong acid?

To 10 c.c. of hydrogen sulphide solution in a test tube add a few drops of lead acetate. (4) What did you observe? (5) For what ion is this experiment a test? (6) Why can not this procedure be used directly as a test for an insoluble sulphide? (7) How may an insoluble sulphide which will react with an acid be tested for? (8) In what recent experiment have you done this?

Put into a test tube a quarter of a spatulaful of copper sulphide, a strip of zinc, and 10 c.c. of hydrochloric acid. Heat the test tube and test for hydrogen sulphide at its mouth. (9) How did you test? (10) What was the result? (11) Write equations for the reactions which took place in the test tube. (12) State the test for a sulphide insoluble in acids.

EXPERIMENT 58. **Sulphides.**

Reference: **282, 283.**

DISCUSSION: The sulphides of the metals fall into three classes, those soluble in water, those which are insoluble but react with dilute acids and are thus said to be soluble in acid, and those which do not react with dilute acid. When hydrogen sulphide is passed into the solution of a salt of the first class, of course no precipitate is formed; when it is passed into a solution of a salt of the second class only part of the metal is precipitated as sulphide, because the reaction is reversible,

since sulphides of this class react with acids formed by the double decomposition; and when it is passed into the third class, complete precipitation of the sulphide takes place. For complete precipitation of members of the second class a soluble salt of hydrogen sulphide must be used in order that no acid shall be formed. These facts are made use of in separating the metals in the course of analysis of salts.

MATERIALS: Solutions of sodium chloride, calcium chloride, zinc sulphate, ferrous ammonium sulphate, copper sulphate, arsenic chloride, hydrogen sulphide, ammonium sulphide.

DIRECTIONS: Put 5 c.c. of solutions of sodium chloride, calcium chloride, zinc sulphate, ferrous ammonium sulphate, copper sulphate, and arsenic chloride into separate test tubes and add 10 c.c. of hydrogen sulphide solution, (either some that you made in Experiment 56, or some of the laboratory reagent) into each.

(1) Tabulate the results of the experiment by writing the names of the compounds in the first column and recording the appearance or non-appearance of precipitates in the second column. (2) Write equations for the formation of precipitates in those cases where they were formed.

Add hydrochloric acid to the tubes containing precipitates until the precipitate disappears or the test tube is full. Record the results in the table in (1). (3) What is the distinguishing characteristic of each of the three classes of sulphides. (4) To which class does each of those just studied belong?

To 5 c.c. of zinc sulphate solution add 10 c.c. of hydrogen sulphide solution, filter, and add 10 c.c. of ammonium sulphide to the filtrate. (5) What was the precipitate finally obtained? (6) Did the hydrogen sulphide completely precipitate the zinc in the first place? (7) Why? (8) How may zinc be completely precipitated as sulphide? (9) How may precipitation of zinc sulphide be entirely prevented when hydrogen sulphide is added?

OXIDES AND ACIDS OF SULPHUR

EXPERIMENT 59. **Formation of Sulphur Dioxide.**

Reference: **287-288.**

DISCUSSION: Sulphur dioxide in many respects resembles carbon dioxide, and like carbon dioxide it may be prepared by the spontaneous decomposition of the acid of which it is the anhydride, and by the oxidation of the element itself. But unlike carbon dioxide it can also be prepared by the reduction of sulphuric acid, to which there is no analogous compound of carbon. The reduction may be considered as the removal of oxygen from the sulphuric acid by the reducing agent and the subsequent decomposition of the resulting sulphurous acid.

MATERIALS: Powdered sulphur, sodium sulphite, copper, powdered charcoal.

DIRECTIONS: (*a*) Touch a bit of sulphur with a hot platinum wire and bring the sulphur thereby into the flame. (**1**) Describe the odor of the substance formed. (**2**) Write the equation for the reaction which takes place when sulphur burns in the air. (**3**) What other element reacts similarly to give an analogous compound?

(*b*) To a few crystals of sodium sulphite in a test tube add dilute sulphuric acid and cautiously smell the gas which is evolved. (**1**) What is it? (**2**) Write two equations to express how the gas was formed. (**3**) Why does this reaction take place? (**4**) What analogous reaction takes place for the same reason?

(*c*) Heat in a test tube half a spatulaful of copper turnings with 5 c.c. of concentrated sulphuric acid and cautiously smell the gas which is formed. (**1**) What is it? (**2**) What element did the copper remove from the sulphuric acid? (**3**) What compound would this leave? (**4**) Into what substances does this compound spontaneously break up? (**5**) How would you

expect the copper compound to react with more sulphuric acid? (6) Why do you predict this action? (7) Write equations for these various reactions and then combine them into one equation. (8) As what kind of an agent did the copper act? (9) The sulphuric acid? (10) What was the product of reduction? (11) What was the product of oxidation? (12) What reaction of double decomposition took place?

(*d*) Repeat experiment (*c*) with the substitution of powdered wood charcoal for the copper. (1) What gas did you recognize as being formed? (2) How did you identify it? (3) From what compound was it formed? (4) By what kind of a reaction? (5) What was oxidized? (6) What was the product of the oxidation? (7) Write equations for the reactions taking place and then combine them into a single equation. (8) What striking difference exists between the final products in this case and those in the action between copper and sulphuric acid? (9) In what respect are the two reactions similar?

(*e*) Repeat experiment (*c*) with the substitution of powdered sulphur for the copper. (1) What gas was formed? (2) What was the final product of reduction of the sulphuric acid? (3) What product did the sulphur form? (4) Write equations for the separate reactions and then combine them into a single equation. (5) Which was oxidized and which reduced? (6) Which was the reducing agent, which the oxidizing agent? (7) What always happens to a reducing agent? (8) To an oxidizing agent? (9) Which of the five methods of formation of sulphur dioxide, which you have just tried, do you consider best adapted for preparing the gas in the laboratory for study? (10) Why?

EXPERIMENT 60. **Preparation of Sulphur Dioxide.**

Reference: 287.

DISCUSSION: As was shown in Experiment 59, sulphur dioxide can be prepared by the action of an acid on a sulphite. A concentrated solution of sodium acid sulphite, however, is much cheaper than sodium sulphite and is usually used in its place. Since a liquid is employed instead of a solid, a slight

change in the form of the gas generator becomes necessary. The presence of the water in the generator requires the use of concentrated sulphuric acid, which is much heavier than the solution. If it were poured down the thistle tube of an apparatus like the one used for preparing carbon dioxide, it would lie as a nearly distinct layer in the bottom of the test tube and not become mixed with the solution of sodium acid sulphite. It is therefore necessary to arrange the apparatus so that the acid will fall through the solution drop by drop.

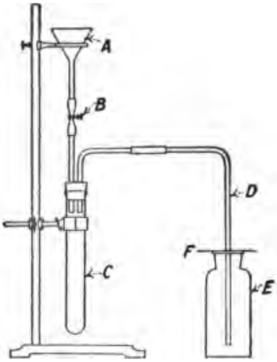

Fig. 12.

Materials: Concentrated solution of sodium acid sulphite, litmus paper.

Directions: Set up an apparatus as shown in Fig. 12. *A* is a funnel supported by a ring and attached by a rubber connector to a tube passing through the stopper in the test tube *C*. The rubber connector must be securely bound with copper wire to the funnel and to the tube and must be long enough to allow the pinch cock *B* to close it completely. *D*, *E* and *F* are the delivery tube, bottle, and filter paper arranged as in former experiments for collecting gases.

Put 15 c.c. of concentrated solution of sodium acid sulphite into the test tube and 10 c.c. of concentrated sulphuric acid into the funnel. By opening the pinch cock slightly allow the acid to flow into the test tube a few drops at a time, so that

a continuous evolution of gas takes place. Moist litmus paper held at the mouth of the bottle will show when it has been filled. After the second bottle has been collected, replace it by a 300 c.c. Erlenmeyer flask one-third full of water, arrange the delivery tube so that the end of it is just above the surface of the water, and continue the evolution of the gas for ten minutes. Cork the flask and keep the solution for Experiments 61 and 62. (1) Write the equations for the reactions taking place in the generator and then combine them into a single equation. (2) Why was the funnel and pinch cock substituted for the thistle tube previously used in the gas generator? (3) Why was concentrated sulphuric acid used instead of dilute acid?

EXPERIMENT 61. **Properties of Sulphur Dioxide, Sulphurous Acid, and Sulphites.**

Reference: **289–294.**

DISCUSSION: The reactions involving sulphur dioxide fall under two heads, namely those in which the valence of the sulphur does not alter and those in which it changes. In the first class the analogy to carbon dioxide is striking, but in the second class no similarity exists, because sulphur unlike carbon can exist in compounds in which it has a valence of six. When sulphur dioxide is converted into a compound in which sulphur exists with the valence of six, the sulphur has been oxidized and hence the sulphur dioxide, by causing oxygen or its equivalent to be given up to the sulphur by the other substance involved in the reaction, has acted as a reducing agent. In contrast to the action of sulphur dioxide as a reducing agent, this substance may also act as an oxidizing agent, when it loses its oxygen and is thereby reduced to uncombined sulphur or still further to hydrogen sulphide.

The bleaching brought about by sulphur dioxide differs chemically from that produced by chlorine, or, more strictly speaking, by hypochlorous acid. The chlorine bleaching consists of the oxidation of the colored substances and this action usually so far destroys the substance that no reversal of the action takes place; that is the bleaching is permanent.

This very completeness of the oxidation, however, precludes this method from use with many fibers, such as silk and wool, because the fibers themselves are easily oxidized and thereby injured by the bleaching agent. On the other hand, sulphur dioxide reduces or unites directly with many colored compounds, producing substances quite similar to the original ones in chemical composition but colorless. Many of these reactions are easily reversible, particularly in the presence of an oxidizing agent, and therefore the bleaching produced by sulphur dioxide is not permanent. Nevertheless the process has the great advantage of not injuring the fiber.

Sulphurous acid and its anhydride sulphur dioxide bear the same relation to each other as was found to exist between carbonic acid and its anhydride, carbon dioxide. No change in valence occurs in the formation of sulphurous acid from sulphur dioxide, so the acid is capable of undergoing the same sort of reactions of oxidation and reduction as its anhydride does.

MATERIALS: Sulphur dioxide prepared in Experiment 60, splinter, copper wire, dyed silk, carnation, potassium chlorate, chlorine water, litmus paper, barium chloride solution, hydrogen sulphide, samples for analysis.

DIRECTIONS: (*a*) (1) What is the color and odor of sulphur dioxide, and its density compared with air?

(*b*) Into one of the bottles of sulphur dioxide thrust a blazing splinter. (1) What happened? (2) What oxide which you have prepared behaves similarly? (3) Would sulphur dioxide be a suitable gas to use as a fire extinguisher? (4) Why?

(*c*) By means of a copper wire suspend a piece of wet pink silk (dyed with rosaniline) and a petal of a carnation in a bottle of sulphur dioxide until a distinct change takes place. (1) What occurred? Rinse the piece of silk under the tap and then boil it in a solution containing a spatulaful of potassium chlorate dissolved in a test tube half full of water. (2) How did the cloth change in appearance? (3) What kind of agent is potassium chlorate? (4) By what kind of reaction was the color restored? (5) By what kind of reaction was it

destroyed? (6) As what kind of an agent did the sulphur dioxide act?

Boil a second piece of dyed silk in a solution of chlorine water. (7) What effect was produced on the color? (8) Was the strength of the fiber of the cloth effected? Rinse the cloth under the tap and boil it in the solution of potassium chlorate. (9) Was the color restored? (10) To what class of reactions does the bleaching process belong? (11) What advantage does it possess over bleaching with sulphur dioxide? (12) What disadvantage?

(*d*) Test the solution into which you passed sulphur dioxide in Experiment 60 with litmus paper. (1) What was the immediate action on the paper? (2) The final action? (3) Write the equation for the reaction between sulphur dioxide and water. (4) Is the reaction reversible? (5) What is the evidence? (6) To what reaction is this analogous?

(*e*) As a preliminary to this experiment make yourself familiar with the test for a sulphate by adding to 5 c.c. of dilute sulphuric acid in a test tube, 5 drops of barium chloride solution and then 5 c.c. of hydrochloric acid. (1) What was the result of adding the barium chloride? (2) Of adding the hydrochloric acid? (3) State the test for a sulphate.

To 5 c.c. of the solution of sulphurous acid prepared in Experiment 60 add 5 c.c. of chlorine water, shake the solution and then test for a sulphate. (4) What was the result of the test? (5) Write the equation for the reaction. (6) What was oxidized? (7) What was the oxidizing agent? (8) As what kind of an agent did the sulphurous acid act?

(*f*) Into a test tube half full of the solution prepared in Experiment 60 bubble hydrogen sulphide from the generator set up in the hood. (1) What insoluble substance was formed? (2) Write the equation for its formation. (3) What was oxidized? (4) What was reduced? (5) What was the oxidizing agent? (6) What was the reducing agent? (7) In summary, write three equations, one in which sulphur dioxide is oxidized, one in which it is reduced, and one in which neither action occurs.

(*g*) (1) State the test for a sulphite based on the facts

brought out in the foregoing experiments. Test the unknown substances given out for the purpose and (2) state whether or not each is a sulphite.

EXPERIMENT 62. **Sulphuric Acid.**

Reference: **305–306.**

DISCUSSION: Sulphuric acid may be formed by the oxidation of sulphurous acid, as was shown in Experiment 61 *e*, or by the oxidation of sulphur dioxide and the subsequent addition of water. In the commercial processes the oxidation is brought about by the oxygen of the air acting directly in the presence of a contact catalytic agent or acting indirectly by the oxidation of an oxide of nitrogen which in turn oxidizes the sulphur dioxide. In the latter method, the higher oxide of nitrogen, having served its purpose as an oxidizing agent and having become thereby reduced, is again oxidized and thus used over and over again. In actual practice the preparation of the sulphuric acid takes place in steps with the preliminary formation of nitrosyl sulphuric acid which is later decomposed. Part (*a*) of this experiment does not reproduce this method of preparing sulphuric acid but it is introduced to show that oxidizing compounds of nitrogen, as nitric acid, will oxidize sulphurous acid to sulphuric acid.

Sulphuric acid has a high boiling temperature; it forms addition products readily, particularly with water to form hydrates; it is an oxidizing agent; and in solution it is highly ionized. These four general properties account for the important chemical reactions which sulphuric acid exhibits.

MATERIALS: Solution of sulphurous acid, concentrated nitric acid, sugar, splinter, cotton cloth, zinc.

DIRECTIONS: (*a*) Test 5 c.c. of the solution prepared in Experiment 60 for sulphuric acid as you tested in Experiment 61 *e*. (1) How did you make the test? (2) What was the result? (3) Is sulphurous acid oxidized rapidly by the air?

To a test tube one-quarter full of the solution of sulphurous acid prepared in Experiment 60 add 10 drops of concentrated nitric acid and boil for two minutes. Dilute with an equal volume of water and test for sulphuric acid. (4) What was

the result? (5) What kind of reaction did the sulphurous acid undergo? (6) What was the agent? (7) Why are special methods of oxidation required in the preparation of sulphuric acid? (8) What are these methods? (9) What is the ultimate source of the oxygen in these methods?

(*b*) (1) Is concentrated sulphuric acid a solution like concentrated hydrochloric acid or is it a pure liquid itself? By means of a glass rod put a drop of concentrated sulphuric acid on a glass plate and nearby put a drop of water. (2) Which spreads out more on the glass? (3) What is the name for sulphuric acid used in the chemical trade and dependent for its origin on this property?

Weigh your empty graduate on the platform scales, pour into it about 10 c.c. of concentrated sulphuric acid, and weigh it again. (4) Record the weights and the volume of the acid. (5) Calculate the specific gravity of the acid.

(*c*) Into 5 c.c. of water in a small test tube pour 5 c.c. of concentrated sulphuric acid and feel the outside of the tube or take the temperature of the mixture with a thermometer. (1) What was your observation? (2) What is the evidence that chemical action takes place between the acid and the water? (3) What is the nature of this action?

To two spatulafuls of sugar ($C_{12}H_{22}O_{11}$) in a test tube add an equal volume of water. Stand the test tube in the rack, add to it 10 drops of concentrated sulphuric acid and if no visible action takes place within one minute add 10 drops more of acid. Cautiously smell at the mouth of the test tube. (4) What is the insoluble substance formed? (5) What has probably happened to the hydrogen and oxygen of the sugar? (6) What property of sulphuric acid caused the action to take place? (7) What substance did you recognize by its odor as being formed? (8) How do you account for its formation?

Put into a test tube a small splinter of wood, a scrap of filter paper, and a small piece of cotton cloth and pour over them a few cubic centimeters of concentrated sulphuric acid. (9) What happenes to each in the course of a few minutes? (10) What do you conclude as to the chemical composition of

these substances? (11) As to the nature of the action of sulphuric acid upon them?

By means of a glass rod make a cross on a piece of filter paper with dilute sulphuric acid. (12) Does any action take place? (13) Why? Dry the paper by moving it back and forth above the flame, taking care that it does not scorch. (14) What happened to the paper? (15) Why did it occur?

(*d*) Heat in a test tube a strip of zinc and 5 c.c. of concentrated sulphuric acid. (1) What insoluble substance was formed? (2) What gas or gases were given off? (3) How did you recognize it or them? (4) What kind of an agent was the acid? (5) What leads you to this conclusion? (6) In what other experiments has sulphuric acid acted as this kind of an agent? (7) What is the relation between the degree of reduction of sulphuric acid by a metal and the position of the metal in the electromotive series? (8) The action of the acid with what two metals illustrates this?

(*e*) (1) What was formed in an earlier experiment by the action of dilute sulphuric acid with zinc? (2) How do you account for the difference between this action and that with concentrated sulphuric acid? (3) What evidence have you already acquired to show that dilute solutions of sulphuric acid contain hydrogen ions? (4) Sulphate ions?

NITROGEN AND THE ATMOSPHERE

EXPERIMENT 63. **Preparation and Properties of Nitrogen.**

Reference: **316–318.**

DISCUSSION: Although nitrogen is the most abundant of the uncombined elements, the removal of the other substances which are mixed with it in the air is so difficult that pure nitrogen is most easily prepared by the decomposition of one of its compounds. If an oxide of nitrogen is chosen as a source, the oxygen is removed by treatment with a reducing agent, as in the reaction between nitric oxide and copper; or if a hydrogen compound of the element is selected, it is treated with an oxidizing agent, as in the reaction between ammonia and copper oxide. The most convenient method, however, should be the preparation from a compound of nitrogen which contains both hydrogen and oxygen in the proportion to satisfy each other, that is in the proportion to form water. Ammonium nitrite, NH_4NO_2, is such a compound and it is easily decomposed by heating. In fact it is so unstable a compound that it is usually prepared only at the instant at which it is to be decomposed. This series of reactions is brought about by heating a mixture of sodium nitrite and ammonium chloride, when the double decomposition between the two substances is immediately followed by the breaking up of the ammonium nitrite into nitrogen and water.

The remarkable lack of chemical activity of nitrogen is easily understood from the fact that nitrogen forms with itself a very stable compound. Although the molecules of a compound are usually thought of as consistnig of atoms of different elements held in chemical combination, molecules of elementary nitrogen likewise consist of atoms held in chemical combination, and may be regarded as a compound of nitrogen with more nitrogen. Since the combination is a very difficult one to

decompose, elementary nitrogen does not readily undergo chemical reaction.

MATERIALS: Ammonium chloride, sodium nitrite, substances necessary to make the tests required in (*b*).

DIRECTIONS: (*a*) Mix thoroughly 5 grams of ammonium chloride with 7 grams of sodium nitrite and place the mixture in a test tube fitted with a thistle tube and delivery tube, as arranged for the preparation and collection of hydrogen in Experiment 8. Have in readiness also a beaker of cold water, which may be held so as to immerse the bottom of the test tube if necessary. Add to the generator 10 c.c. of water and warm the test tube gently. As soon as gas begins to be formed, remove the burner and if the action becomes so vigorous that the contents of the tube begins to froth, hold the beaker of cold water so that it will cool the tube until the action becomes less violent. A few cubic centimeters of water may also be poured down the thistle tube to decrease the frothing if it continues. Collect two bottles of nitrogen. **(1)** Write equations for the two reactions taking place in the generator. **(2)** Why is not ammonium nitrite itself used for the preparation of nitrogen? **(3)** Of what radicals is ammonium nitrite composed? **(4)** In the decomposition of the compound, what element does the nitrogen of the ammonium radical lose? **(5)** What is the removal of this element from a compound termed? **(6)** What element did the nitrogen of the nitrite radical lose in the decomposition? **(7)** What is the loss of this element termed? **(8)** Why should the composition of ammonium nitrite make it particularly suitable for the preparation of nitrogen? **(9)** Was the decomposition exothermic or endothermic? **(10)** What was the experimental evidence?

(*b*) **(1)** State the properties of nitrogen as to odor, color, and solubility in water. **(2)** From what common knowledge might you have predicted these properties without preparing pure nitrogen?

Try experiments when necessary with the nitrogen which you have collected, such as will enable you to answer the following questions. In each case state what you did to prove your answer correct. **(3)** How can nitrogen be distinguished

from oxygen? (4) From hydrogen? (5) From chlorine? (6) From hydrochloric acid? (7) From sulphur dioxide? (8) What do you consider the most striking chemical property of nitrogen? (9) What conclusion do you draw as to the stability of the nitrogen molecule? (10) Why do you draw this conclusion?

EXPERIMENT 64. **The Atmosphere. Percentage of Oxygen.** Reference: **321.**

DISCUSSION: Air is a mixture, not a pure compound and, therefore, the proportion of its constituents is not constant. The proportion of oxygen, however, in samples taken from different localities varies only slightly and is usually expressed in per cent by volume. This percentage is determined by removing the oxygen from a measured volume of air and observing the volume of the gas which remains, or the volume of oxygen absorbed. Since the volumes of gases change considerably with changes of temperature and changes of pressure, care must be taken that the initial and final measurements of volumes are taken under the same conditions.

In the following determination, air confined in a flask of known volume is shaken with a solution of sodium pyrogallate, which absorbs the oxygen, thus causing a partial vacuum in the flask. The flask is now opened under water and a volume of water will flow in equal to the volume of the oxygen which has been absorbed. The percentage of oxygen in the air is then calculated from the volume of oxygen and the original volume of air in the flask.

MATERIALS: Solutions of pyrogallic acid (10 per cent), and of sodium hydroxide (50 per cent); screw pinchcock.

DIRECTIONS: The apparatus consists of a 300 c.c. Erlenmeyer flask fitted with a one-hole rubber stopper into which is thrust a short piece of glass tubing flush with the bottom of the stopper and attached at the other end to a stout rubber connector provided with a screw pinchcock. Care must be taken that the stopper and tubes fit tightly.

Fill the flask completely with water, loosen the pinchcock, push the stopper into the flask as far as it will readily go,

close the pinchcock, remove the stopper, and measure the volume of water in the flask by means of your graduate. (1) Record the volume.

Fill a pneumatic trough, adding enough hot water to bring the temperature to that of the air in the room. (2) Why?

Pour into the flask 15 c.c. of the special solution of pyrogallic acid and 30 c.c. of the special solution of sodium hydroxide, each measured accurately in the graduate, and *immediately* insert the stopper in the flask and close the pinchcock. At once put the neck of the flask under water in the pneumatic trough and keep it there until the experiment is finished. (3) Why is the neck of the flask kept under water? Shake the flask at frequent intervals for fifteen minutes. (4) What is taking place in the flask during this period?

Hold the flask on its side in the water and loosen the screw of the pinchcock, taking care to keep the end of the rubber tube *constantly below* the surface. After the water has stopped running in, remove the flask from the trough, measure carefully in your graduate the volume of the liquid in the flask, and (5) record the value.

The original volume of air is equal to the volume of the flask as obtained in the beginning of the experiment minus 45 c.c., and the volume of the oxygen is equal to the final volume of liquid in the flask minus 45 c.c. (6) Why must 45 c.c. be subtracted in the first case? (7) In the second case? (8) In calculating the percentage of oxygen, state why it is, or is not, necessary to take into account the barometric pressure; (9) the temperature of the air. (10) Calculate from your data the percentage of oxygen in the air. (11) What is the actual percentage? (12) What is the ratio of your error to the actual percentage?

EXPERIMENT 65. **The Quantitative Determination of Carbon Dioxide in Exhaled Air.**

Reference: **322.**

DISCUSSION: The amount of carbon dioxide present in a sample of air can be determined quantitatively by shaking a known volume of the sample with a solution of barium hy-

droxide of known strength, and observing how much of the latter is required to convert the carbon dioxide present into barium carbonate. The reaction is carried out in the presence of an indicator in order to determine when an excess of the hydroxide has been added. Exhaled air contains 4 per cent by volume of carbon dioxide.

MATERIALS: One 2-liter bottle, a 10 c.c. graduated pipette, solid $Ba(OH)_2,8H_2O$, phenolphthalein.

DIRECTIONS: To obtain a solution of barium hydroxide of known concentration place in a test tube 2 grams of crystalline barium hydroxide and 20 c.c. of water, the temperature of which is approximately 20°. Close the tube with a cork and shake vigorously for 3 minutes. At the end of this time there should be present some crystals of the hydroxide which have not dissolved. Set the tube aside to allow the solid to settle.

Practice in the following way the use of a 10 c.c. pipette until you can deliver from it any volume desired. Moisten the index finger of the right hand and place the upper end of the pipette in the mouth and the lower end below the surface of some water. Draw up the water by suction until the level is above the zero point of the graduation. Place the index finger over the upper end. Slightly release the pressure of the finger until the liquid falls, drop by drop, from the pipette. To stop the flow increase the pressure of the finger. Practice delivering 1 c.c. of water.

Determine the exact volume of a 2-liter bottle by filling it with water from a graduate. (1) Record this volume and also the temperature of the water and the barometric pressure. Insert the bottle filled with water in a pneumatic trough, and then completely replace the water in the bottle with air from the lungs by blowing into it through a rubber tube. Cork the bottle and remove it from the trough. Remove the cork, and place in the bottle 10 c.c. of water and about 5 drops of a solution of phenolphthalein. Replace the cork.

(2) Record the temperature of the solution of barium hydroxide. Without disturbing the solid on the bottom of the tube fill the pipette with the solution to the line slightly above the zero point. (The presence of a small amount of suspended

barium carbonate in the solution will not affect the results.) Let the solution run back into the tube until the level of the liquid is just at the zero point, and then let 10 c.c. of it run into the bottle of air. Cork the latter tightly and shake it vigorously until the pink color of the indicator has disappeared (about 2 minutes). Add 1 c.c. of the barium hydroxide solution and shake again. Continue the addition of the barium hydroxide, 1 c.c. at a time, until a deep pink color remains after shaking the bottle vigorously for 3 minutes. **(3)** Record the total number of cubic centimeters added. (If in the addition of the base slightly more or less of it has been added as the result of inability to stop the flow from the pipette at the exact point, record the exact volume added.)

The weights of $Ba(OH)_2$ contained in 1 c.c. of a saturated solution at different temperatures are as follows: 18°, 0.035 g.; 20°, 0.037 g.; 22°, 0.040 g. Calculate from the number of cubic centimeters used and the concentration of the solution at the temperature of the experiment **(4)** the weight of $Ba(OH)_2$ required to react with the CO_2 present in the air; **(5)** the weight of CO_2 equivalent to this ($Ba(OH)_2 + CO_2 = BaCO_3 + H_2O$); **(6)** the volume of this weight of CO_2 when measured at the temperature and pressure of the air in the bottle; and **(7)** the total volume of air in the bottle, after correcting for the liquid added. **(8)** What is the percentage by volume of CO_2 in the exhaled air examined? **(9)** By how much does this value differ from what has been determined by exact experimentation?

A man exhales about 12 cubic meters of air per day. Calculate what **(10)** volume and **(11)** weight of CO_2 is exhaled per day. **(12)** What is the weight of carbon in this weight of CO_2? The carbon required is obtained from the food. If sugar, which contains 42 per cent of carbon were the food, what weight would be required to furnish the necessary carbon?

EXPERIMENT 66. **Dew Point. Relative Humidity.**

Reference: **94–96, 323.**

DISCUSSION: For every temperature there exists a definite

maximum concentration of water vapor beyond which condensation will take place if an excess of vapor is present. The concentration is expressed as the pressure of this vapor in terms of millimeters of mercury. This maximum concentration or pressure is the same as the vapor pressure of water at the particular temperature in question. For example, the maximum vapor pressure of water vapor at 10° is 9.2 mm. and at 20° is 17.4 mm.

The pressure of water vapor in the air may be determined by cooling the air and observing the temperature at which condensation takes place. Thus, if condensation begins at 10°, the pressure of the water vapor in the air is 9.2 mm. but if condensation begins at 20°, the pressure must be 17.4 mm. The temperature at which condensation takes place is called the dew point. By observing the dew point and referring to a table of vapor pressures at different temperatures, the concentration or pressure of the water vapor in the air may be found.

If the air has to be cooled in order to cause condensation, it is obvious that the air could have contained more water vapor at the original temperature. The maximum pressure of water vapor which could exist at this original temperature may be found by again referring to the table. The relation between this maximum pressure and the actual pressure as shown by the dew point is spoken of as the relative humidity of the air. To illustrate: the air on a certain day was at a temperature of 25° and it was found necessary to cool it to 16° before the dew point was reached. By reference to the table, on page 542, it appears that the vapor pressure at 16° is 13.5 mm. and at 25° is 23.6 mm. Thus the actual vapor pressure was 13.5 mm., the maximum vapor pressure which could have existed at 25° was 23.6 mm. and the relative humidity on that day was $13.5/23.6 = .57 +$ or 57 per cent.

To find the dew point experimentally, ice may be added slowly to water in a beaker, the outside of which has been carefully cleaned, and the temperature at which condensation begins on the outside of the beaker may be noted. Since it is very difficult to see the very first film of moisture, too low

a temperature is usually recorded. If now the ice is removed from the beaker and the water inside allowed to rise in temperature, the condensed water will evaporate and should disappear when the temperature rises above the dew point. But here too there is usually a "lag" and too high a temperature is found. The average between the temperature of appearance of the film and the temperature of its disappearance will be very close to the true dew point.

MATERIALS: Ice.

DIRECTIONS: Test the accuracy of the graduations on your thermometer as directed in Experiment 51. (1) What correction do you find it necessary to make?

Clean and polish the outside of a 200 c.c. beaker, fill it about one-third full of tap water and place it so the light from the window comes to your eye by reflection from the side of the beaker.

Put into the beaker a lump of ice, about 10 c.c. in volume, stir the water with your thermometer, and watch the outside of the beaker for the appearance of a film of moisture. If the ice melts before the film appears add more ice, lump by lump, as it melts, until you see the condensed water. (1) Record the temperature at which the film appears.

Remove the ice, continue the stirring, and (2) note the temperature at which the film disappears. Again determine and record the temperature at which the film appears by adding ice, and at which it disappears when the ice is removed. (3) Correct the recorded temperatures by the amount of the error of your thermometer and obtain the average of these four values. (4) What is this temperature called?

Wipe the thermometer and hang it on a ring stand on your desk. When the mercury has ceased to rise, (5) record the observed temperature and the corrected temperature of the air.

Referring to the table of vapor pressures of water on page 542, (6) record the vapor pressure for the dew point which you have found and for the temperature of the air. (7) What relation has the first value to the pressure of the water vapor actually existing in the air? (8) What is the maximum pressure of water vapor possible in the air at the existing tem-

perature? (9) What is the ratio of the pressure of the actual vapor present to the pressure of the possible maximum? (10) What is this value expressed in per cent? (11) What is this percentage called?

AMMONIA

EXPERIMENT 67. **Sources of Ammonia.**

Reference: **336–343.**

DISCUSSION: Formerly all ammonia was derived from organic nitrogen compounds which were decomposed by heating them. Those compounds occurring in coal were the chief source and still are. The ammonia formed in this way is always mixed with large quantities of other substances formed during the decomposition of the original material. When the gaseous mixture is passed into the solution of an acid, the ammonia forms a compound with the acid, which may be obtained as a solid upon evaporation of the solution. This ammonium compound will again yield ammonia in a fair state of purity, when treated with a base.

Ammonia is the only gas of common occurrence which forms an alkaline solution with water and its presence may thus easily be detected with wet litmus paper.

MATERIALS: Gelatine, ammonium sulphate, concentrated solution of sodium hydroxide, powdered sodium hydroxide, powdered sodium nitrate, powdered iron, calcium cyanamide, litmus paper.

DIRECTIONS: (*a*) Heat in a dry test tube a spatulaful of gelatine and try the action of the gas formed upon a piece of wet pink litmus paper held at the mouth of the tube. (1) What was the action on the paper? (2) For what gas is this a test? (3) Of what kind of substances is this action of gelatine typical?

(*b*) Refer to your notes on Experiment 42. (1) What is the evidence that ammonia is given off upon heating coal? (2) Why must ammonia from this source be further treated before being used?

(*c*) To half a spatulaful of ammonium sulphate on a watch

glass add a few drops of concentrated sodium hydroxide and observe the odor of the gas given off. (1) What was the gas? (2) What is the evidence that it is in a purer state than the ammonia obtained directly from coal? (3) How can ammonium sulphate be prepared from ammonia? (4) By what series of steps can pure ammonia be obtained from coal?

(*d*) Mix thoroughly half a spatulaful of powdered sodium hydroxide (handle this with care as it is very corrosive) with an equal amount of powdered sodium nitrate and 2 spatulafuls of powdered iron. Heat the mixture in a test tube and test for the evolution of ammonia. (1) How did you test and with what result? (2) By what sort of reaction was ammonia formed in this experiment?

(*e*) Boil in a test tube a mixture of half a spatulaful of calcium cyanamide and 10 c.c. of water. (1) Was ammonia formed? (2) What did you do in order to answer question (1)? (3) Write the equation for the reaction which took place. (4) Write two equations to show how calcium cyanamide is made.

EXPERIMENT 68. **Preparation and Properties of Ammonia.**
Reference: **336, 344–346.**

DISCUSSION: The preparation of ammonia from ammonium chloride and calcium hydroxide resembles the preparation of carbon dioxide from an acid and a carbonate in that a double decomposition is immediately followed by the spontaneous breaking up of one of the products into water and a gas. In both cases also the secondary reaction is reversible and with ammonia this reversal, together with the great solubility of ammonia gas itself, is so marked as to require the use of solid material instead of solutions, in order to reduce as much as possible the amount of water present.

Ammonia adds directly to the acids and forms a series of compounds resembling the salts. Just as oxygen and a nonmetal may form the negative part of a salt, which exists only as an ion in solution and can not be obtained uncombined, so four atoms of hydrogen and one of nitrogen may form the positive part of a salt, which likewise exists only as an ion and

has not been obtained uncombined. This radical or ion is formed by the direct union of ammonia with the hydrogen of an acid, but upon attempting to decompose the compound to obtain the uncombined radical, the reaction of formation is reversed and the original ammonia and acid are obtained.

MATERIALS: Ammonium chloride, calcium hydroxide, splinter, litmus paper, concentrated hydrochloric, and nitric acids.

DIRECTIONS: (*a*) Set up an apparatus as shown in Fig. 13. *A* is a *dry* 250 c.c. bottle resting in a clamp *B*. *C* is a glass tube extending nearly to the top of the bottle and projecting about 1 cm. below the bottom of the stopper in the 8-inch test tube *D*.

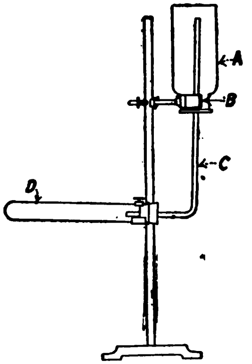

FIG. 13.

Mix 10 grams of ammonium chloride with 10 grams of calcium hydroxide in a mortar and put the mixture into the test tube. Holding the burner in your hand, heat the test tube gently beginning at the point marked *D* in the figure and slowly work forward. Test for ammonia at the mouth of the bottle with wet litmus paper and continue the evolution of the gas at least one minute after a test is obtained. Remove the bottle, and cover it with a glass plate, keeping it inverted all the time. Collect two more bottles of ammonia in the same way.

(1) Write two equations for the reactions taking place in the generator and then combine them into a single equation. (2) Which of these reactions is easily reversible? (3) Why are

the solid materials used instead of solutions of them? (4) Name substitutes which might have been used instead of ammonium chloride and calcium hydroxide. (5) Write equations for the reactions which would have taken place had these substitutes been used. (6) Why was the bottle held in an inverted position? (7) Why should the delivery tube extend nearly to the top of the bottle?

(*b*) Thrust a burning splinter into one of the bottles of ammonia. (1) What was the result? (2) How does this compare with the similar experiment that you tried with the hydrogen compound of sulphur?

(*c*) Hold one of the bottles of ammonia mouth downward in the water in the pneumatic trough, shake the bottle keeping the mouth under water, replace the glass plate, and remove the bottle from the trough. (1) What occurred when the bottle was opened under water? (2) What property of ammonia caused this action? (3) What was the gas remaining in the bottle? (4) How did it get there?

Drop a piece of pink litmus paper into the water in the bottle. (5) What was the action on the litmus? (6) For what ion is this action on litmus a test? (7) Does either water or ammonia contain this ion? (8) Does ammonia react chemically with water? (9) State the logical steps upon which you base your answer. (10) Name the four substances beside water which exist in a solution of ammonia.

(*d*) Into the third bottle of ammonia thrust a glass rod which has been wet with a concentrated solution of hydrochloric acid. (1) What did you observe? (2) Write an equation for the reaction which occurred.

Repeat the experiment using concentrated nitric acid on the rod instead of hydrochloric acid and again using dilute hydrochloric acid and concentrated sulphuric acid. (3) What did you observe in each case? (4) What is the reason for the difference in the results?

(*e*) Thrust the end of a piece of glass tubing about 20 cm. in length into a one-hole rubber stopper. Put into the tube enough ammonium chloride to occupy about 1 cm. of its length and by tapping and rolling the tube get the material near the

middle of it. Clamp the stopper to a ring stand so that the tube is inclined as much as is possible without allowing the ammonium chloride to slip out. Heat the substance and hold pieces of both kinds of wet litmus paper at each end of the tube. (1) What happened, and (2) what was the action due to in each case? (3) Write an equation for the reaction which took place upon heating the ammonium chloride. (4) Why was the experiment carried out in an inclined instead of horizontal tube? (5) What was deposited near the ends of the tube? (6) Why was it deposited instead of passing out? (7) By what reaction was it formed? (8) What will happen if ammonium chloride is heated in an open dish? Try the experiment by heating a little in a porcelain dish. (9) Was your prediction verified?

EXPERIMENT 69. **The Determination of the Percentage of Nitrogen in Ammonia.**

Reference: **345–346.**

DISCUSSION: When ammonia is treated with a solution of bleaching powder, $CaOCl_2$, (**116**), the chlorine of the latter unites with the hydrogen of the ammonia and nitrogen is set free. In the experiment described below, the amount of ammonia in a solution is determined by finding out how much hydrochloric acid of known strength is required to neutralize 20 c.c. of the solution. The amount of nitrogen in 20 c.c. of the solution is obtained in the way indicated above. From the two results the percentage of nitrogen in ammonia can be calculated.

The required solution of hydrochloric acid of known strength is made by diluting concentrated hydrochloric acid, the composition of which must first be determined.

MATERIALS: Concentrated hydrochloric acid, methyl orange, bleaching powder.

DIRECTIONS: (*a*) To determine the composition of the concentrated hydrochloric acid, weigh your graduated cylinder to decigrams, pour into it about 10 c.c. of the acid, weigh again and (1) record the weights and also the volume to 0.1 c.c. (2) Calculate the density of the acid.

The percentages by weight of hydrochloric acid in solutions of different densities are as follows: density 1.17, 33.5 per cent; 1.18, 35.4 per cent; 1.19, 37.2 per cent. (3) What percentage of hydrochloric acid did your solution contain? (4) How many grams of the pure compound were contained in the solution you weighed out?

Add water to the acid in the graduate up to the 50 c.c. mark, pour the solution into a dry beaker and add to it 150 c.c. more of water. (5) Calculate from the weight of acid obtained in (4) the weight in 1 c.c. of this diluted solution.

(*b*) To exactly 20.0 c.c. of dilute ammonia (5 normal) add exactly 80.0 c.c. of water in a flask, shake the solution, pour exactly 20.0 c.c. of it into a beaker, and add to it 3 drops of a solution of methyl orange, to serve as an indicator. Put exactly 50.0 c.c. of the dilute hydrochloric acid solution prepared in (*a*) into a graduated cylinder and add it little by little to the ammonia until the indicator just changes from yellow to red. (1) Record the number of cubic centimeters used. This is only a preliminary trial. Repeat the neutralization, adding nearly the required amount of acid quite rapidly and making the final addition drop by drop. (1) Record the volume of acid used to 0.1 c.c. (2) From the value obtained in (*a*) 5, calculate the weight of acid in this volume; (3) the weight of ammonia required to react with this weight of acid ($NH_3 + HCl = NH_4Cl$). (4) What is the weight of ammonia in 20 c.c. of your solution?

(*c*) Fit a 250 c.c. flask with a stopper carrying a thistle tube and a delivery tube arranged to collect a gas over water. Put in the flask 10 grams of fresh bleaching powder and 100 c.c. of water. Insert the stopper and place the delivery tube under an inverted 250 c.c. bottle filled with water in a pneumatic trough. Add to the flask exactly 20 c.c. of your ammonia solution and then 10 c.c. of water. Shake the flask occasionally. When gas is no longer evolved, cover the gas bottle with a plate and remove it from the trough. Pour in additional water from your graduate to fill the bottle completely and (1) record the volume added, the temperature of the water, and the barometric pressure. (2) Subtract from this volume 30 c.c.

and state what this value represents. (3) Why was the subtraction made? (4) Calculate the volume of the nitrogen under standard conditions, not forgetting to take into account the vapor pressure of the water. One cubic centimeter of nitrogen under standard conditions weighs 0.00125 gm. (5) Calculate the weight of nitrogen contained in the 20 c.c. of ammonia solution which you used. (6) From this value and from the weight of ammonia obtained in (*b*) 4 calculate the percentage of nitrogen in ammonia. (7) Calculate from the formula the true percentage.

ACIDS AND OXIDES OF NITROGEN

EXPERIMENT 70. **Preparation and Properties of Nitric Acid.**
Reference: **357, 360–366.**

DISCUSSION: Since nitric acid boils at a much lower temperature than sulphuric acid, the double decomposition between a nitrate and sulphuric acid will proceed, if the temperature is high enough to make the nitric acid pass off as fast as it is formed. Nitric acid, however, readily decomposes with the formation of a brown gas, when it is heated; for this reason the acid is prepared at the lowest temperature which can be used to effect the reaction. As a consequence only the first step in the double decomposition, which involves the formation of an acid sulphate, takes place; the second step, producing a normal sulphate, requires a higher temperature.

Nitric acid is so corrosive that it attacks rubber or cork stoppers and it thus becomes necessary to conduct the preparation of the acid in an apparatus composed entirely of glass.

It has already been shown, in Experiment 62 on the oxidation of sulphurous acid to sulphuric acid, that nitric acid is a strong oxidizing agent, and the study of the action of nitric acid with a variety of substances further emphasizes this important property. Since the reduction products of nitric acid usually are brown gases or gases which turn brown in the air, it is easy to recognize, as such, a reaction in which nitric acid is acting as an oxidizing agent.

MATERIALS: Sodium nitrate, sulphur, solution of barium chloride, retort, sand bath.

DIRECTIONS: PRECAUTION: *Nitric acid is very corrosive. Use great care not to get a drop of it on your skin or clothing.* (*a*) Set up an apparatus as shown in Fig. 14. *A* is a retort clamped to a ring-stand and resting on an iron pan, *D*, filled

with sand. The neck of the retort extends into an 8-inch test tube, *B*, which stands in a beaker, *C*, partly filled with cold water.

Slip into the retort by means of a creased filter paper 12 grams of sodium nitrate. Place a funnel in the opening of the retort and through it pour in 10 c.c. of concentrated sulphuric acid. Insert the stopper of the retort and slowly heat the sand in the pan. When about 5 c.c. of nitric acid have distilled into the test tube, remove the flame, and raise the clamp which holds the retort several inches on the ring stand, so that the test tube may be slipped off and the retort allowed to cool, away from the sand. Save the nitric acid for subsequent experiments.

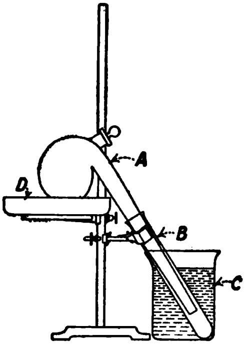

FIG. 14.

When the retort has become quite cool, fill it about half full of warm water and let it stand until the solid which it contains is somewhat dissolved and becomes loosened from the glass. The contents of the retort may be poured into the sink and washed down the waste pipe with a large amount of water from the tap.

(1) Write an equation for the reaction by which the nitric acid was formed. (2) Write the equation for another reaction which may take place between sodium nitrate and sulphuric acid. (3) How could the second reaction be caused to take place? (4) Why is this not done? (5) How does your nitric

acid differ in appearance from the concentrated nitric acid in the laboratory? (6) What is the cause of this difference? (7) What substances could be used instead of sodium nitrate in this preparation? (8) Could hydrochloric acid be substituted for sulphuric acid? Why? (9) Why is this form of generator used instead of the simpler one used in preparing hydrochloric acid?

(*b*) Pour about 1 c.c. of the nitric acid which you have prepared into a 6-inch test tube standing in the test tube rack. Be careful not to let the acid run down the outside of either test tube. Hold the smaller test tube in a holder and heat the acid cautiously. (1) What was formed? (2) Write the equation for the reaction.

(*c*) Dilute the remainder of the acid which you prepared with an equal volume of water. If you do not have enough acid to perform the following experiments, the concentrated nitric acid in the laboratory may be used instead.

To about 3 c.c. of your acid in a small test tube add a small pinch of sulphur and boil until the sulphur disappears. Add enough water to half fill the test tube and then test the solution for a sulphate. (1) How did you make the test? (2) What was the evidence that sulphuric acid was present? (3) What property of nitric acid does this experiment show? (4) State how it proves it? (5) Write three partial equations to show how the nitric acid decomposes, how the sulphur is oxidized, how sulphuric acid is formed from the product, and then combine these equations into a single one. (6) Why should a different reaction of decomposition of nitric acid be written in this case from that written for the decomposition in (*b*)?

EXPERIMENT 71. **Nitrates.**

Reference: **367–368.**

DISCUSSION: It has already been shown in the preceding experiment that nitric acid is decomposed by heat. Nitric acid, however, is the nitrate of hydrogen and it is important to know whether the nitrates of other elements also decompose in the same way. This experiment is designed to answer this

question. Sodium nitrate is chosen as an example of the nitrates of very active metals, of which group potassium is the only other common metal, and copper nitrate as an example of the nitrates of less active metals. It might be expected that ammonium nitrate, being the nitrate of a radical instead of a metal, might behave differently from all other nitrates. The action of ammonium nitrate will be studied more fully in Experiment 73.

In testing for a nitrate advantage is taken of the fact that a very small amount of nitric oxide will form a dark brown compound with ferrous sulphate, $FeSO_4,NO$. As has already been shown nitric acid is reduced to nitric oxide by certain reducing agents, of which ferrous sulphate is one. In other words, a dark brown compound is formed when nitric acid is added to ferrous sulphate, because the latter first reduces the nitric acid and then unites with the nitric oxide produced. Thus the formation of this compound may serve as a test for nitric acid. The test may be made applicable not only to nitric acid but also to any nitrate by the addition of concentrated sulphuric acid, since the sulphuric acid will convert the nitrate, if present, to nitric acid, which will in turn react with the ferrous sulphate as just described. In actual practice the sulphuric acid is added last and in such a way that it will form a layer in the bottom of the test tube, because it is heavier than the other liquids. The reactions will then take place where the layers meet and the brown compound will appear as a ring, if a nitrate is present. It must be remembered, however, that nitrous acid and the nitrites also produce nitric oxide, and consequently the brown compound, under these circumstances. The test is of value, therefore, only when nitrites are known to be absent.

MATERIALS: Sodium nitrate, copper nitrate, solutions of sodium nitrate, sodium nitrite, and ferrous sulphate.

DIRECTIONS: (*a*) Heat a few crystals of sodium nitrate in an ignition tube. It is necessary to heat the substance to as high a temperature as possible. When a gas is seen to be vigorously given off, hold a glowing splinter at the mouth of the tube. (1) What was the action? (2) What did this

prove the gas to be? (3) Was any nitrogen dioxide given off? (4) What is the evidence? (5) Does sodium nitrate decompose in the same way as hydrogen nitrate? (6) If part, but not all, of the element which you identified were given off upon heating the sodium nitrate, what compound would remain? Test the truth of your answer by letting a drop of concentrated sulphuric acid run down into the ignition tube after it has cooled. (7) What happened? Try the action of a drop of concentrated sulphuric acid on a crystal of the substance named in the answer to question 6. (8) Was your answer to question 6 proved to be correct or not? (9) How? (10) Write an equation for the reaction taking place upon heatsodium nitrate. (11) What other nitrate behaves in the same way?

(*b*) Heat a few crystals of copper nitrate in a small test tube and continue the heating until no further change takes place. (1) What gas was given off? (2) How did you recognize it? (3) What remained in the test tube? (4) By what property did you recognize it? (5) Write an equation for the reaction of decomposition. (6) How does the decomposition resemble that of nitric acid? (7) Why was an ignition tube required for the heating of sodium nitrate, whereas a test tube could be used for copper nitrate?

(*c*) To 5 c.c. of sodium nitrate solution in a test tube add an equal volume of ferrous sulphate solution. Holding the test tube inclined, carefully pour down its side 3 c.c. of concentrated sulphuric acid, which should form a distinct layer in the bottom of the tube. (1) What happened between the two layers? (2) What is the formula of the compound? (3) What two functions does the ferrous sulphate serve? (4) Why was the sulphuric acid added? (5) Why was the test tube inclined when the acid was added? (6) State the test for a nitrate.

Repeat the experiment with the substitution of a solution of sodium nitrite for sodium nitrate. (7) What is the observed result? (8) Can a nitrate be tested for in the presence of a nitrite? (9) Why?

EXPERIMENT 72. **Nitrous Acid and Nitrites.**

Reference: **369–373.**

DISCUSSION: Nitrous acid is even less stable than nitric acid. This is shown from the fact that when the salts of the acids are treated with sulphuric acid at room temperature, the nitrate produces nitric acid which is only slightly decomposed, whereas the nitrite forms nitric oxide, nitric dioxide, and water instead of nitrous acid.

Nitrous acid, like sulphurous acid, may be reduced and also oxidized, or in other words may act as an oxidizing agent or as a reducing agent.

MATERIALS: Solution of sodium nitrite, iodine, sodium iodide, potassium permanganate, starch.

DIRECTIONS: (*a*) To 5 c.c. of sodium nitrite solution in a test tube add 5 c.c. of dilute sulphuric acid. (**1**) What occurred? (**2**) What compound did you recognize as being formed? (**3**) What nitrogen compound would you expect to be formed by double decomposition? (**4**) What do you conclude as to the stability of nitrous acid? (**5**) Formulate a test for a nitrite. (**6**) How can a nitrite be distinguished from a nitrate?

(*b*) As a preliminary to this experiment it is necessary to become familiar with a test for iodine. Grind in your mortar with 5 c.c. of water a lump of starch the size of a small pea, put the paste into a large test tube half full of water and boil until the liquid no longer looks cloudy. Dilute 5 c.c. of iodine solution with 10 c.c. of water and add a few drops of this diluted iodine to 5 c.c. of the starch emulsion. (**1**) What color appeared? This is a test for iodine. (**2**) State the test for iodine. Add a few drops of sodium iodide solution to 5 c.c. of starch emulsion. (**3**) What occurred? (**4**) Can starch be used as a test for uncombined iodine as well as for iodine alone?

To 5 c.c. of starch emulsion add a single drop of sodium iodide solution, 5 c.c. of dilute sulphuric acid, and finally 5 c.c. of sodium nitrite solution. (**5**) What happened? (**6**) What was thus proved to have been formed in the reaction? (**7**) Write an equation for the reaction by which it was formed,

assuming that nitrous acid and hydrogen iodide had previously been formed by the action of sulphuric acid on the sodium nitrite and sodium iodide respectively. (8) What kind of reaction did the hydrogen iodide undergo? (9) What was the agent? (10) What sort of reaction did the nitrous acid undergo?

(*c*) When potassium permanganate is reduced the characteristic purple color disappears.

To 5 c.c. of water in a test tube add 3 drops of potassium permanganate solution, 5 c.c. of dilute sulphuric acid and 5 c.c. of sodium nitrite solution. (1) What change in color occurred? (2) What happened to the nitrous acid present? (3) State in logical sequence the steps by which you arrived at this conclusion? (4) What compound which you have already studied resembles nitrous acid in that it can be both reduced and oxidized?

EXPERIMENT 73. **Nitrous Oxide.**

Reference: **376–377.**

DISCUSSION: Just as the hydrogen and oxygen of ammonium nitrite were found in Experiment 63 to unite to form water when the compound was heated, so the hydrogen and oxygen of ammonium nitrate act when this compound is heated. But a molecule of ammonium nitrate contains one more atom of oxygen than the number required to react with the hydrogen present, so the resulting residue is nitrous oxide, N_2O, instead of elementary nitrogen, N_2, as in the case of the nitrite.

Nitrous oxide decomposes into a mixture of nitrogen and oxygen at a temperature below that at which most substances burn. Hence when a glowing splinter is thrust into nitrous oxide a mixture is formed which is richer in oxygen than is the air, and combustion therefore proceeds more vigorously than in the air. Moreover nitrous oxide is an endothermic compound, that is it evolves heat upon decomposition, and the additional heat thus furnished still further increases the rate of combustion.

MATERIALS: Ammonium nitrate, splinters.

DIRECTIONS: Set up an apparatus consisting of an 8-inch

test tube provided with a one-hole rubber stopper through which passes a delivery tube leading to a pneumatic trough. Have in readiness a beaker of cold water with which to check the reaction if it becomes too rapid, as you did in Experiment 63. Fill the pneumatic trough and gas bottles with water at about 40°. Put 10 grams of ammonium nitrate in the test tube, insert the stopper, and heat with a low flame until the ammonium nitrate melts and the nitrous oxide is slowly evolved. Collect two bottles full of the gas and immediately disconnect the delivery tube from the generator. (1) Why? (2) Write an equation for the reaction taking place in preparing the nitrous oxide. (3) What very similar reaction have you recently studied?

(*b*) Thrust a glowing splinter into one of the bottles of nitrous oxide. (1) What occurred? (2) With what gas does a similar reaction take place? (3) What does this experiment suggest about the stability of nitrous oxide upon heating? (4) How does it indicate it? (5) Write an equation for the reaction of decomposition of nitrous oxide. (6) Would you expect combustion to take place more or less vigorously than in air? (7) Why? (8) What did you actually find to be the case? (9) Was the total heat evolved in burning the splinter in nitrous oxide derived from the oxidation of the wood? (10) Upon what property of nitrous oxide, stated in the discussion, do you base your answer?

Open the second bottle of nitrous oxide inverted under cold water in the pneumatic trough. (11) What happened? (12) What property of nitrous oxide is shown by this action? (13) Why was the gas collected over warm rather than cold water?

EXPERIMENT 74. **Nitric Oxide, Nitrogen Dioxide, and Nitrogen Tetroxide.**

Reference: **379–382.**

DISCUSSION: Nitric oxide and nitrogen dioxide have already been met as reduction products of nitric acid. In preparing nitric oxide an agent of the proper reducing power and nitric acid of the proper concentration must be used or else other products of the reduction of nitric acid will be formed instead.

Copper and concentrated nitric acid to which has been added an equal volume of water are found to be the most convenient substances to produce nitric oxide in a fair degree of purity. It must be collected out of contact with the air because it unites directly with oxygen. Since it is much more stable than nitrous oxide, it will not support combustion, as does the latter.

In the relation of nitrogen dioxide, NO_2, to nitrogen tetroxide, N_2O_4, there is exemplified a kind of equilibrium not heretofore emphasized in these experiments, namely that which may exist in some cases between single molecules and double or poly molecules of the same percentage composition. Nitrogen tetroxide, which is colorless, is converted into nitrogen dioxide, which is brown, as the temperature is raised. At room temperature both gases exist together in equilibrium.

In the reactions of nitrogen tetroxide and of nitrogen dioxide with water is illustrated the oxidation of part of a compound at the expense of the remainder, which is thus reduced. Nitrogen exhibits the valence of four toward oxygen in nitrogen dioxide and tetroxide, but when the latter reacts with water nitric acid and nitrous acid are both formed, in which compounds nitrogen shows the valences of five and three respectively. That is, some of the nitrogen has been oxidized from valence four to valence five and some reduced from four to three. At a higher temperature more is oxidized to nitric acid; consequently that which is reduced must be further reduced than in the first case; that is, nitric oxide, in which nitrogen has the valence of only two, is formed instead of nitrous acid.

MATERIALS: Copper turnings, concentrated nitric acid, lead nitrate, ice.

DIRECTIONS: (*a*) Arrange an apparatus like the one used in Experiment 8 for the preparation and collection of hydrogen. Put 10 grams of copper turnings in the test tube. Dilute 10 c.c. of concentrated nitric acid with 10 c.c. of water and pour the mixture down the thistle tube. If the reaction becomes too rapid, a little cold water may be added and the outside of the generator cooled as in previous experiments. Collect two bottles of gas. (1) What was the color of the gas in the

generator immediately after the action began? (2) What was the color of the gas collected? (3) What is its name? (4) Write an equation for the reaction by which it was formed. (5) Why was it necessary to use diluted nitric acid? (6) Why was copper rather than a much more active metal chosen for this preparation?

(*b*) Thrust a burning splinter into a bottle of nitric oxide. (1) What occurred? (2) How is the difference between this action and that of nitrous oxide in respect to combustion accounted for? (3) What did you observe upon opening the bottle of nitric oxide? (4) Write an equation for the reaction. (5) How do you account for the changes in color in the gases in the generator during the preparation of nitric oxide? (6) Can nitric oxide be collected by displacement of air? (7) Why?

(*c*) Uncover the second bottle of nitric oxide, and pour into it 25 c.c. of water, immediately cover it tightly with the palm of your hand, and shake it. (1) What two occurrences show that the nitrogen dioxide formed upon opening the bottle is absorbed by the water? Let a little more air into the bottle and again shake it. After doing this several times, test the water with litmus paper. (2) What was the action? (3) Write an equation for the reaction between the gas and the water. (4) In forming nitric acid from the gas, what sort of a process has the nitrogen undergone? (5) What was the agent? (6) What sort of a change did the agent undergo? (7) What did it form? (8) What changes in valence of nitrogen are involved in this reaction? (9) What would have been formed at a considerably higher temperature?

(*d*) Put a spatulaful of lead nitrate into an 8-inch test tube and clamp the tube in a horizontal position. Fit it with a one-hole rubber stopper through which passes a delivery tube, bent at right angles and extending to the bottom of a small test tube supported in a gas bottle. Heat the lead nitrate until two test tubes have been filled with the gas. Cork the tubes and place one tube in a gas bottle full of hot water and the other in a bottle full of ice and water. (1) What difference in color is observable in the two samples of gas? (2) To what is this difference due? (3) What compound is chiefly present

in the cold tube? (4) In the hot tube? (5) Write an equation to show the relation of these gases to each other.

Allow both tubes to come to room temperature by standing them in the test tube rack. (6) Is any difference of color finally observable? (7) What is now present in the tubes?

ATOMIC AND MOLECULAR WEIGHTS

EXPERIMENT 75. **The Molecular Weight of a Gas.**

Reference: **388–390.**

DISCUSSION: The volume occupied by one gram-molecular-weight of a gas under standard conditions is 22.4 liters. Hence if the weight of 22.4 liters is known, the molecular weight is known. On account of the difficulty of handling so large a volume of gas as 22.4 liters, the weight of a measured volume is found and the weight of 22.4 liters is computed.

MATERIALS: Kipp carbon dioxide generator with apparatus for drying the gas.

DIRECTIONS: Clean and dry a 500 c.c. flask, select a tightly fitting cork, and weigh the flask and cork to centigrams. **(1)** Record the weight. Pass carbon dioxide, prepared in a Kipp generator and dried by passing through sulphuric acid, into the flask until it overflows as shown by the action on a burning match held near the mouth of the flask. The carbon dioxide delivery tube should extend to the bottom of the flask. **(2)** Why? When the flask has been filled, cork it and weigh it again. Record the weight under **1.** Again pass carbon dioxide into the flask and weigh it. **(3)** If there is a gain in weight upon the second addition of gas, what do you conclude about your first attempt to fill the flask? Continue to successively pass in carbon dioxide and weigh the flask, until you are sure that you have the weight when it is full. **(4)** How will you know when you have obtained this weight?

Weigh on the platform balance and **(5)** record the weight of the flask full of water and corked. **(6)** What is the volume of the flask? **(7)** Record the temperature and the barometric pressure. One liter of air under standard conditions weighs 1.293 grams. **(8)** What is the weight of air in the flask? **(9)** Of the flask empty? **(10)** Of the carbon dioxide? **(11)** What

would be the volume of the carbon dioxide under standard conditions? (**12**) What would be the weight of 22.4 liters? (**13**) What do you find the molecular weight of the gas to be? (**14**) What is the true molecular weight? (**15**) What error in the molecular weight would be caused by an error of a centigram in weighing the flask when filled with carbon dioxide? (**16**) How great an error in weighing the flask full of water would have caused the same error in your final result?

EXPERIMENT 76. **Determination of the Molecular Weight of Carbon Tetrachloride.**

Reference: **388–389.**

DISCUSSION: The molecular weight of a gas is determined by weighing a sample of the gas at any temperature and pressure and calculating with the aid of the gas laws what weight of the gas would occupy 22.4 liters at 0° and 760 mm. pressure, provided the behavior of the gas with change in temperature and pressure was that indicated by the laws of gases.

The experiment given below illustrates a method that can be used when only approximate results are desired. The substance used is carbon tetrachloride, CCl_4. It is a liquid at ordinary temperatures (boiling point, 76.6°), but exists as a gas at the temperature of boiling water. The molecular weight of carbon tetrachloride in the gaseous condition is determined by heating some of the liquid in a weighed flask of known volume placed in boiling water until the flask contains only the vapor of the compound. The flask is next cooled to room temperature. The vapor liquefies and air enters the flask to take its place. The flask is weighed; the increase in weight is the weight of the known volume of the vapor of carbon tetrachloride at the temperature observed and at the pressure of the atmosphere. From the weight of the vapor and its volume reduced to 0° and 760 mm. the weight of 22.4 liters under these conditions can be calculated. This value is the molecular weight.

MATERIALS: A flask which holds approximately 300 c.c. when completely filled, large beaker as high as the flask used, copper wire, carbon tetrachloride.

Directions: Fit the dry clean flask with a cork stopper containing two holes. In one place a thermometer so that the bulb of the latter will be about 3 to 4 cm. below the stopper. Pass through the second hole a piece of glass tubing about 3 to 4 cm. long, one end of which is drawn out so that the diameter of the opening is about 1 mm. The wide end of the tube should project below the cork about 0.5 cm. Place the stopper carrying the thermometer and glass tube in the flask and (1) weigh to decigrams on the platform scales.

Measure about 5 c.c. of carbon tetrachloride in a dry cylinder and pour it into the flask. Place the flask in the beaker and wire it down so that it will stay in place when water is added to the beaker. This can be done conveniently by placing a piece of copper wire around the beaker just below the top, having one long end and one short end. Wind the short end around the wire itself and bring the long end over the stopper between the thermometer and the glass tube. Next bend the wire drawn tight over the edge of the beaker.

Place the beaker on a stand, and pour in boiling water until it is three-fourths full. Heat the water to boiling. Wind a towel around the thermometer and let it rest on the beaker. Take care that the ends of the towel do not hang over the beaker and come in contact with the flame. The towel serves to retain the steam in the upper part of the beaker and thus secures a constant temperature in the flask.

The carbon tetrachloride boils and escapes from the flask through the glass tube. A small amount condenses at first, however, in the latter and drops back into the flask. Continue the heating for about 10 minutes after the liquid ceases to drop. (The total time for the heating is about half an hour.) (2) Note the temperature recorded on the thermometer. Remove the flask from the water. Dry it carefully with a towel and let it stand 15 minutes. (3) Weigh the flask with the stopper, thermometer and glass tube to decigrams. The difference between this weight and that of the empty flask is the weight of the vapor at the temperature observed. To determine the volume of the flask pour out the carbon tetrachloride, fill the flask completely with water. and insert the

stopper carrying the thermometer and glass tube. Remove the stopper and (4) measure with a graduated cylinder the volume of the water that remains in the flask. The volume of the water is equal to that of the vapor contained in the flask. (5) By the use of the gas law formula reduce this volume to 0° and 760 mm. This volume of vapor has the weight found in the experiment. (6) From these two figures calculate the volume of 22.4 liters. (7) What is the molecular weight of carbon tetrachloride as calculated from its formula? (8) How great was your error? (9) What was your percentage error? (10) Why was no correction made for the weight of air in the flask?

EXPERIMENT 77. **Determination of the Atomic Weight of Manganese.**

Reference: **392.**

DISCUSSION: The determinations of the atomic weights of metallic elements are usually made by converting one compound of the metal into another compound. If the atomic weights of the other elements in the compounds are known, it is possible to calculate the atomic weight of the metal. In the experiment described below the atomic weight of manganese is determined by converting manganese carbonate, $MnCO_3$, into manganous oxide, MnO:

$$MnCO_3 = MnO + CO_2$$

A weighed quantity of manganese carbonate is heated. The loss in weight is the weight of the carbon dioxide given off. The difference between this and the weight of the carbonate used is the weight of the manganous oxide. The calculation of the atomic weight is made by solving the following proportion: Molecular weight of manganous oxide (= atomic weight of manganese + 16) : molecular weight of carbon dioxide (= 44) = observed weight of manganous oxide: observed weight of carbon dioxide, or

$$Mn + 16 : 44 = \text{wt. } MnO : \text{wt. } CO_2$$

Materials: Manganese carbonate.

Directions: (1) Weigh a crucible and cover to centigrams. Place in it from 2.5 to 3 grams of manganese carbonate and weigh to centigrams. Support the crucible and cover on a triangle and heat cautiously by moving the flame of a burner under the crucible. At the end of about one minute place the burner under the crucible and have the flame of such a size that it reaches to the cover. Do not remove the cover until the crucible is cold. The carbon dioxide given off from the carbonate prevents the oxidation of the manganous oxide. If the latter is heated to redness in air it changes to Mn_3O_4. Heat for 10 minutes. Allow the crucible to cool for 10 minutes and (1) weigh. (2) Calculate the atomic weight of manganese in the way indicated in the discussion above. (3) What is the true atomic weight? (4) How great was your error? (5) What per cent of the true atomic weight was your error? (6) By what other method have you determined the atomic weight of a metal?

Experiment 78. **Determination of the Atomic Weight of Copper.**

Reference: **392–393.**

Discussion: In determining the atomic weight of a metal a rough approximation of the value can be calculated from the specific heat of the metal by applying Dulong and Petit's law, which states that the atomic weight multiplied by the specific heat = 6.4. The accurate value is obtained by the analysis of a compound of the metal. In the experiment described below the atomic weight of copper is calculated from the weight of copper obtained from a known weight of copper sulphate, $CuSO_4,5H_2O$. A solution of copper sulphate is treated with finely divided zinc. The reaction which takes place is indicated by the following equation:

$$CuSO_4 + Zn = ZnSO_4 + Cu$$

The excess of zinc is dissolved from the product by means of dilute hydrochloric acid, which does not affect the copper. Taking 32 as the atomic weight of sulphur, 16 as that of oxygen,

and 1 for hydrogen, the atomic weight of copper can be calculated by solving the following proportion: Weight of $CuSO_4$, $5H_2O$ taken : Weight of Cu formed = At. wt. Cu + 32 + 4 (16) + 5 (18) : at. wt. Cu.

MATERIALS: Copper sulphate, zinc dust, alcohol.

DIRECTIONS: (1) Weigh to decigrams on the platform scales approximately 20 grams of copper sulphate, $CuSO_4,5H_2O$. The crystals should be clear blue and not covered with a white deposit. Place the salt in a 250 c.c. beaker, add 100 c.c. of water, heat, and stir until the salt has dissolved. Cool the beaker in running water, and add cautiously in small portions 7 grams of zinc dust, which has been rubbed with a glass rod so that it contains no lumps. Stir during the addition of the zinc. In about 5 minutes add in small portions 10 c.c. of concentrated hydrochloric acid. Place the beaker on a wire gauze and boil gently for 30 minutes. Remove the beaker and stir to determine if hydrogen is still evolved from the excess of zinc used.

When the gas is no longer given off, add 100 c.c. of water, heat to boiling, remove the beaker from the gauze, let the copper settle and pour off most of the liquid through an 11 cm. filter paper. Add 100 c.c. of water to the copper, heat to boiling, let the copper settle, and decant off the liquid as before. Remove by means of a spatula as much of the copper as possible to the filter paper. With the aid of a little water transfer the rest of the copper to the filter paper. Heat about 50 c.c. of water to boiling and pour it through the filter to complete the washing of the copper. If alcohol is available the drying of the copper can be effected more rapidly. Pour alcohol on the copper up to the edge of the paper. When no liquid drops from the funnel wash again with alcohol. Remove carefully the paper containing the copper from the funnel, and place the former in a dry evaporating dish. Place the dish in a drying closet or on the steam bath. When the paper is thoroughly dry, weigh it and the copper to centigrams. Place on the other pan of the balance a piece of filter paper of the same size. The weight observed is the weight of the copper. Record this weight under **1**.

(2) Calculate the atomic weight of the metal in the way indicated in the discussion above. (3) Calculate an approximate atomic weight of copper from the fact that its specific heat is 0.091. (4) Which method of determination gives the more accurate result? (5) Why? (6) Of what value, then, is the other method? (7) In this experiment is it necessary to weigh the zinc accurately? (8) Why?

THE HALOGENS

EXPERIMENT 79. **Preparation and Properties of Bromine.**

Reference: **401–403.**

DISCUSSION: Fluorine, chlorine, bromine, and iodine show striking similarity in their chemical properties, and they all fall into the same column of the periodic classification of the elements. In studying such a group, or so-called "family," of elements, one may be studied in detail and then the differences and resemblances between the members of the family may be emphasized. Thus bromine should be constantly compared with chlorine, and iodine in turn with these two.

MATERIALS: Potassium bromide, manganese dioxide.

DIRECTIONS: PRECAUTION: *Bromine is very corrosive and its vapor attacks the membrane of the nose and throat. Perform all experiments involving bromine in the hood.* Grind in a mortar about 2 grams of potassium bromide, add an equal volume of manganese dioxide and transfer the mixture to an 8-inch test tube, clamped to a ring stand and fitted with a one-hole stopper through which passes a delivery tube leading to the bottom of a small test tube placed in a bottle of cold water. To 5 c.c. of water add 3 c.c. of concentrated sulphuric acid (never add the water to the acid) and when the mixture has cooled, pour it into the test tube which contains the potassium bromide mixture and at once replace the stopper in the test tube. Heat the mixture until a few drops of liquid bromine have been collected in the receiver. When the preparation of the bromine has been completed, empty the generator into the bottle provided for the purpose.

(1) Write equations for the two reactions involved in the preparation of bromine and then combine the equations into a single one. (2) What other member of this group of elements have you studied? (3) What elements are included in the

group? (4) In what respects does the preparation of bromine resemble or differ from the preparation of chlorine? (5) What is the color of bromine vapor? (6) Is the vapor heavier or lighter than air? (7) What is the evidence? (8) Calculate from the atomic weight of bromine, which is 80, the density of bromine vapor compared with air, the density of which is the same as that of a gas having the molecular weight of 28.8. (9) What is the color of liquid bromine? (10) Is it a comparatively volatile liquid? (11) What is the evidence? (12) How does it compare with chlorine in this respect? Pour 5 c.c. of water into the test tube containing the bromine. (13) What is the density of liquid bromine compared with water? (14) What is the evidence as to its solubility? Drop a small piece of litmus paper into the bromine and water. (15) What was the action on the litmus? (16) What other element acts in the same way?

Pour the bromine and water into the bottle provided to receive it.

EXPERIMENT 80. **Hydrobromic Acid.**

Reference: **405–407.**

DISCUSSION: When a bromide is treated with concentrated sulphuric acid, hydrobromic acid and an acid sulphate are formed, as would be expected; but then a further reaction takes place. Just as hydrochloric acid can be oxidized by a sufficiently strong oxidizing agent, as was shown in Experiment 23, so hydrobromic acid also may be oxidized. But this latter compound is more readily oxidized than hydrochloric acid and even sulphuric acid, which does not react with hydrochloric acid, serves to bring about this oxidation. Hence the hydrobromic acid expected from the double decomposition of a bromide and sulphuric acid will contain bromine and the reduction products of the sulphuric acid. Pure hydrobromic acid may be prepared by the substitution of phosphoric for sulphuric acid or by some entirely different method, such as the reaction between phosphorus pentabromide and water.

MATERIALS: Potassium bromide.

DIRECTIONS: Place two or three crystals of potassium

bromide in an evaporating dish and pour on them a few drops of concentrated sulphuric acid. (1) What color does the liquid become? (2) To what is the color due? (3) Write equations to show by what reactions it was formed. (4) Does a chloride behave in a similar way when treated with sulphuric acid? (5) Which is the stronger reducing agent, hydrochloric or hydrobromic acid? (6) What is the evidence?

Blow across the top of the evaporating dish. (7) What was the result? (8) With what compound did you once observe a similar action? (9) The presence of what compound is indicated by the phenomenon in this case? (10) Explain how this phenomenon proves that hydrobromic acid is very soluble in water. (See Experiment 25.) (11) How can pure hydrobromic acid be prepared?

EXPERIMENT 81. **Iodine.**

Reference: **411–413.**

DISCUSSION: The preparation of iodine is similar to the preparation of chlorine and bromine.

At room temperature iodine is a very volatile solid, that is it has a high vapor pressure. Upon heating it, the vapor pressure increases and reaches atmospheric pressure at a temperature below the melting point. Hence it sublimes instead of melting, as was explained in **186.** The chemical activity of iodine is less than that of the other halogens but yet it will oxidize many compounds and is extensively used for this purpose, particularly in quantitative chemical analysis. An important reaction in these processes is that between iodine and sodium thiosulphate.

$$2Na_2S_2O_3 + I_2 = 2NaI + Na_2S_4O_6$$

MATERIALS: Potassium iodide, manganese dioxide, alcohol, carbon disulphide, chloroform, solutions of potassium iodide, of sodium acid sulphite, of sodium thiosulphate, starch.

DIRECTIONS: (*a*) Grind in a mortar about 2 grams of potassium iodide, add an equal volume of manganese dioxide and put the mixture in a crucible on a wire gauze supported on

a ring of the ring stand. Moisten the material with water and add 3 drops of concentrated sulphuric acid. Cover the crucible with a watch glass, into which pour a little water to keep it cool. Heat the crucible gently until the bottom of the watch glass becomes coated with crystals of iodine. When the crucible has cooled, empty it into the bottle provided for the purpose.

(1) Write equations for the two reactions involved in the preparation of iodine and then combine the equations into a single one. (2) To what group of elements does iodine belong? (3) Why did the iodine vapor condense to a solid as it was formed instead of first passing through the liquid state? (4) Describe the appearance of solid iodine.

(*b*) Transfer a few crystals from the watch glass to a test tube and heat them. When the tube is full of vapor incline it mouth downward. (1) What is the color of iodine vapor? (2) Is it heavier or lighter than air? (3) What is the evidence? (4) Calculate the density of iodine vapor compared with air. (The molecular weight of iodine is $2 \times 127 = 254$.)

(*c*) Put a small crystal of iodine into each of five test tubes and add to them respectively 5 c.c. of water, of alcohol, of potassium iodide solution, of carbon disulphide, and of chloroform. (1) What were the colors of the solutions? (2) Is iodine as soluble in water as in the other liquids? (3) What is the evidence? (4) What is the evidence that iodine reacts with potassium iodide? (5) Write the equation for the reaction.

(*d*) Make some starch emulsion as directed in Experiment 28 *d* and to 10 c.c. of it add a drop of the water solution of iodine. (1) What color appears? Now add sodium acid sulphite solution until the color disappears. (2) What does the disappearance of the color show concerning the iodine? (3) Write an equation which shows what has happened to it. (4) As what kind of an agent has it acted? Repeat the experiment using a solution of sodium thiosulphate instead of sodium acid sulphite. (5) Write an equation for the reaction which took place. (6) Under what circumstances is this reaction used?

EXPERIMENT 82. **Hydriodic Acid.**

Reference: **415–417.**

DISCUSSION: Just as hydrobromic acid can be prepared by the action of water on phosphorus bromide, so hydriodic acid can be prepared from water and phosphorus iodide. The phosphorus iodide can be made by the direct union of phosphorus and iodine.

As was pointed out in Experiment 80, hydrobromic acid is a more powerful reducing agent than hydrochloric acid, being able to reduce sulphuric acid to sulphurous acid. From the position of iodine in the periodic classification, it would be predicted that hydriodic acid would be a still more powerful reducing agent than hydrobromic acid, and that further reduction products of sulphuric acid might be expected, when an iodide is treated with sulphuric acid. Whether or not this prediction is verified is shown in part (*b*) of this experiment.

MATERIALS: Iodine, red phosphorus, potassium iodide.

DIRECTIONS: (*a*) Place one crystal of iodine, 2 or 3 mm. in diameter, on not more than twice its volume of red phosphorus in an evaporating dish. Let one drop of water from the end of a glass rod fall upon the mixture. Marked action will usually take place within a minute. If it does not, the bottom of the dish may be warmed very slightly. Phosphorus tri-iodide is formed. (1) Write an equation for the reaction. After the reaction is completed, add another drop of water and blow across the top of the dish. (2) What appeared? (3) To what compound is the action presumably due? (4) What is the reason for this belief? (5) Write an equation for the reaction between the water and the phosphorus iodide.

(*b*) Pour a few drops of concentrated sulphuric acid on a crystal of potassium iodide on a watch glass. Observe closely the substances formed in the dish and smell the gases given off by wafting them toward you with your hand. (1) What reduction products of sulphuric acid did you recognize? (Three are usually detectable.) (2) What further evidence was there of the oxidation of hydriodic acid? (3) Was any hydriodic acid given off? (4) How did you test for it? (5) Write an equation for the first reaction taking place between potassium

iodide and sulphuric acid, and three equations for the formation of the reduction products of sulphuric acid. (6) Is hydriodic acid or hydrobromic acid the more powerful reducing agent? (7) What is the evidence? (8) From what fact could you have predicted their relative reducing power?

EXPERIMENT 83. **Hydrofluoric Acid.**

Reference: **424, 426–428.**

DISCUSSION: Hydrofluoric acid is too corrosive and poisonous a substance to be prepared by an elementary class in sufficient quantities to observe its physical properties. Enough can be made, however, to show its action on glass, which is one of its most important chemical reactions.

Fluorine is the most electronegative, or non-metallic, of the elements, as would be anticipated from its position at the top of group VII of the periodic classification. Consequently it forms stable compounds, not only with the metals, but even with such non-metals as silicon. Thus, a reaction of double decomposition takes place between hydrofluoric acid and the oxide of silicon, just as between hydrochloric and the oxide of a metal.

It is convenient to regard calcium silicate, $CaSiO_3$, as a compound of calcium oxide and silicon dioxide (CaO,SiO_2), for thus it is evident why hydrofluoric acid acts on calcium silicate to form the fluorides of both calcium and silicon, together with the second product of double decomposition, water. Glass is a mixture of silicates and silicon dioxide, and when it is exposed to hydrofluoric acid it is acted upon in accordance with the reactions just described. As the action takes place unevenly on a polished surface of glass, the surface becomes pitted and produces a frosted or etched appearance.

MATERIALS: Calcium fluoride, paraffin.

DIRECTIONS: PRECAUTION: *Exercise great care not to inhale any hydrofluoric acid or allow it to touch your skin. Work in the hood.* Warm a glass plate by holding it high above the flame, where it will not get so hot as to crack. When the glass is warm enough to melt paraffin, rub a lump of this wax over one side of the plate so as to form a thin even coating. Scratch

some design through the wax with the end of a file, being careful that the paraffin is entirely removed and the glass exposed along the lines.

To 5 grams of calcium fluoride in a lead dish add just enough concentrated sulphuric acid to form a paste when thoroughly stirred with a file. Place the glass plate, wax side down, over the dish. At the end of half an hour remove the plate and at once scrape the contents of the dish into the bottle prepared to receive it and then wash the dish thoroughly.

Scrape the paraffin from the glass plate with a knife and observe the action of the hydrofluoric acid on the glass. (1) What has happened to the glass? (2) Why was it coated with paraffin? (3) Write an equation for the reaction between the calcium fluoride and sulphuric acid. (4) Was there any evidence of the reduction of sulphuric acid, as in the case of treating sodium iodide with sulphuric acid? (5) Why could you have predicted this result? (6) Write equations for the reactions taking place between the hydrofluoric acid and (*a*) the silicon dioxide, (*b*) the calcium silicate, and (*c*) the sodium silicate in the glass. (7) What do these reactions show concerning the chemical nature of fluorine? (8) Explain why the glass appears etched instead of smooth where it was exposed to the hydrofluoric acid. (9) Describe a test for a fluoride.

EXPERIMENT 84. **Comparisons of the Halogens and of the Halides.**

Reference: **441.**

DISCUSSION: In the following comparisons the fluorides are omitted because fluorine itself is so difficult to prepare.

The relative reducing power of the halogen acids has been discussed in Experiment 82 and the relative oxidizing power of the halogens themselves necessarily follows this property in the reverse order. Closely associated with the relative oxidizing power is the order in which one halogen may replace another from a solution of a compound.

Valuable tests for the indentification of the halogens depend upon the insolubility of the silver halides and their relative tendencies to form soluble complex compounds with ammonia.

MATERIALS: Solutions of sulphurous acid, chlorine, bromine, barium chloride, sodium chloride, potassium bromide, potassium iodide, sodium carbonate, silver nitrate.

DIRECTIONS: (*a*) (1) Name the halogen acids, except hydrofluoric acid, in the order of their reducing power. (2) Upon what experimental evidence do you base this answer? (3) At which end of the series would you expect hydrofluoric acid to stand? (4) Why? (5) What experiment accords with this prediction?

(*b*) (1) In what experiment did you determine the relative replacing power of the halogens? (2) What did you find the order to be? (3) What was this series then called? (4) To what property of the elements was the replacing power stated to be due? (5) In respect to what other property do the halogens follow the same order?

(*c*) To 5 c.c. each of solutions of sodium chloride, potassium bromide and potassium iodide add 3 c.c. of dilute nitric acid and then a few drops of silver nitrate solution. (1) State what happened in each case, including the color produced. (2) Write equations for the reactions, both in the molecular and ionic forms.

Add to each test tube ammonium hydroxide, about 5 c.c. at a time, shaking after each addition, until the precipitate disappears or the solution contains a large excess of ammonia as indicated by the odor. (3) What happened in each case? (4) How can the three halides be distinguished from one another? (5) In the case of the disappearance of a silver halide upon adding ammonia, what kind of a compound was formed?

To 5 c.c. of sodium carbonate solution add a few drops of silver nitrate and then an excess of nitric acid. (6) What happened after the addition of each reagent? The action of nitric acid upon the precipitate in this case may be taken as typical of the action on all insoluble silver salts other than the silver halides. (7) Formulate a test for the halides in general. (8) Why is it necessary to use nitric acid in this test? (9) State a test for a chloride, (10) two tests for a bromide and (11) three tests for an iodide.

EXPERIMENT 85. **Hypochlorites and Chlorates.**

Reference: **431–433.**

DISCUSSION: The action of chlorine on water with the formation of hydrochloric and hypochlorous acids was discussed under chlorine in Experiment 22. If the water contains a base in dilute solution, the acids react to form their salts and a mixture of chloride and hypochlorite is formed.

Bleaching powder, which in the solid state is a mixed salt (calcium chloride-hypochlorite), is commonly used for bleaching. A solution containing sodium hypochlorite, sometimes called Javelle water, is often more convenient and can be prepared by treating bleaching powder with sodium carbonate and filtering.

As was pointed out in the discussion of the oxides of nitrogen, Experiment 74, a compound which is capable of both reduction and oxidation may sometimes decompose of itself, part being oxidized at the expense of the remainder. Hypochlorites offer another example of this kind of reaction, where part of the hypochlorite is oxidized to chlorate and part reduced to chloride. If chlorine is passed into a hot concentrated solution of a base, a chlorate and a chloride are formed instead of hypochlorite and chloride as in the case of a cold dilute solution.

MATERIALS: Bleaching powder, sodium carbonate, colored cloth, potassium hydroxide, chlorine, splinter.

DIRECTIONS: (*a*) (1) In what experiment which you have performed was hypochlorous acid formed? (2) Write an equation for the reaction which took place. (3) What action can be used as a test for hypochlorous acid?

(*b*) To 10 grams of bleaching powder in an evaporating dish add enough water to make a thin paste. Place two strips of colored cloth in the paste and keep a third for a standard of comparison. Remove one of the pieces of cloth from the paste and dip it into a beaker of dilute sulphuric acid, again cover it with the bleaching powder, and again dip it in the acid. Repeat this four or five times and then wash both pieces of cloth under the tap and compare them with the original and with each other. (1) What changes have

taken place and to what extent? (2) How can bleaching powder be treated to make it a more rapid bleaching agent?

(*c*) Mix thoroughly in your mortar a spatulaful of bleaching powder and one of sodium carbonate. Put the mixture into a large test tube, add 20 c.c. of water, shake the tube for several minutes, and filter. Put a small piece of colored cloth into 5 c.c. of the filtrate. (1) What happened to the piece of cloth? (2) Write an equation for the reaction between the sodium carbonate and the bleaching powder. (3) What is the commercial name of the filtrate?

(*d*) Dissolve 3 grams of solid potassium hydroxide in 7 c.c. of water in a test tube and while the solution is still warm bubble into it chlorine from the generator in the hood until crystals are precipitated. Cool the tube under the tap and filter out the crystals, keeping them in the tip of the cone of filter paper. Remove the filter from the funnel, spread it out, and carefully transfer the crystals to a sheet of dry filter paper. When the substance is thoroughly dry put some of it into an ignition tube and heat it. Be careful that no shreds of filter paper get into the tube. Test at the mouth of the tube for oxygen. (1) How did you test? (2) With what result? (3) What were the crystals? (4) Write equations in five steps and a combined equation to show how the substance was formed.

PHOSPHORUS, ARSENIC, ANTIMONY AND BISMUTH

EXPERIMENT 86. **Phosphoric Acids and Phosphates.**

Reference: **458–463.**

DISCUSSION: When the oxide of a non-metal reacts with water, the resulting hydroxide is an acid. It is theoretically possible to form a large number of different acids from the same oxide and water by causing reactions to take place between the two in different proportions. In the cases which have already been studied of the action of carbon dioxide, sulphur dioxide, and sulphur trioxide upon water only one of the possible acids has been formed in each case, because the others are unstable or do not exist at all. With phosphorus, however, three well-defined acids are known which may be regarded as being formed from one molecule of phosphorus pentoxide and one, two, and three molecules of water, respectively. The formulas for these acids are therefore P_2O_5,H_2O or HPO_3, $P_2O_5,2H_2O$ or $H_2P_2O_7$, and $P_2O_5,3H_2O$ or H_3PO_4. They are all called phosphoric acid, but are distinguished by the prefixes meta-, pyro-, and ortho- respectively. These three acids form corresponding salts which differ in their properties and can thus be made use of in tests for the three different acid radicals. Orthophosphoric acid introduces a further complexity, in that three sets of salts may be derived from it, namely those in which one, two, or three hydrogen atoms have been replaced by the same metal. These are called primary, secondary, and tertiary orthophosphates, respectively.

When phosphorus pentoxide, or as it is commonly called phosphoric anhydride, is put into cold water, the meta acid is formed. Upon boiling this slowly it reacts with more water and produces the ortho acid. The pyro acid which might be expected as an intermediate product is not formed. This acid, or more usually one of its salts, can be prepared, however, by

the reverse process, namely by the dehydration of a secondary ortho compound. The dehydration of a primary orthophosphate gives a metaphosphate.

MATERIALS: Disodium orthophosphate, microcosmic salt, phosphoric anhydride, solutions of ammonium molybdate, silver nitrate, calcium chloride.

DIRECTIONS: (*a*) Preparation of a pyrophosphate. Heat gently in a porcelain crucible 2 grams of disodium orthophosphate. Gradually increase the heat to the highest temperature obtainable and continue the heating for 10 minutes. When the crucible has cooled, dissolve the contents in cold water and keep the solution for the tests in part (*c*). (1) Write an equation for the reaction. (2) Of what acid is the product a salt? (3) What is the relation of this acid to phosphoric anhydride?

(*b*) Preparation of a metaphosphate. Repeat experiment (*a*) with the substitution of microcosmic salt, $NaNH_4HPO_4$, for the disodium phosphate, saving the solution for the tests in (*c*). Test the gas given off with red litmus paper. (1) What substance do you recognize as a product of decomposition? (2) Write an equation for the reaction. (3) A salt of what acid is formed? (4) What is the relation of this acid to phosphoric anhydride? (5) Does microcosmic salt decompose like a primary or like a secondary orthophosphate? (6) Upon what do you base your answer?

(*c*) Tests. (1) Prepare a table for recording the results of the following tests by writing in the first column the names, ortho, pyro, meta, by heading the second column molybdate, the third silver nitrate, and the fourth calcium chloride. Record in this table the formation and color of precipitates when they appear. To 1 c.c. of a solution of disodium orthophosphate add 2 c.c. of dilute nitric acid, 3 c.c. of ammonium molybdate and warm the solution until a precipitate appears.

Into each of two test tubes put 5 c.c. of a solution of disodium orthophosphate and add to one a few drops of silver nitrate solution and to the other a few drops of calcium chloride solution. Repeat the foregoing tests with the pyrophosphate and metaphosphate solutions prepared in (*a*) and (*b*). (2)

What is a general test for all phosphates? (3) How can each of the three phosphates be distinguished from the other two?

(*d*) Drop a quarter of a spatulaful of phosphoric anhydride into 15 c.c. of cold water in a beaker and stir the solution. (1) Describe the appearance of phosphoric anhydride. (2) By what other name is it known? (3) What was the evidence of vigorous reaction betwen the oxide and water? Determine by testing a small amount of the solution which phosphoric acid was formed. (4) What test did you use and with what result? (5) What acid was present? (6) Write an equation for its formation.

To the remainder of the solution add 5 c.c. of dilute nitric acid, which acts only as a catalyst, and boil it for 10 minutes. (7) Which acid is now present? (8) Upon what tests do you base this answer? (9) Write an equation for the formation of the acid. (10) Name in order the three compounds which may be regarded as successive stages of hydration of phosphoric anhydride.

EXPERIMENT 87. **Halides of Phosphorus.**

Reference: **467.**

DISCUSSION: Phosphorus tri-iodide has already been prepared by the direct union of phosphorus and iodine in Experiment 82. Two sets of halides would be expected corresponding to the two valences in which phosphorus appears in combination. All of these compounds are known with the exception of phosphorus penta-iodide. Since phosphorus is a distinctly acid-forming rather than base-forming element, its halides may be completely hydrolyzed in water.

MATERIALS: Phosphorus trichloride, phosphorus pentachloride.

DIRECTIONS: *Perform these experiments in the hood.* (*a*) Pour a few drops of phosphorus trichloride into a *dry* test tube. (1) Describe the appearance of the compound. Blow across the mouth of the test tube. (2) What occurred? (3) What do you conclude is being formed?

Half fill the test tube with water. (4) What phenomenon do you observe? (5) Explain its cause. (6) Write an equation for the reaction between phosphorus trichloride and water.

(*b*) Examine a small amount of phosphorus pentachloride, about 1 gram. (1) Describe its appearance. Determine whether or not it will hydrolyze. (2) What did you do and with what results? (3) What do you conclude? (4) What property of phosphorus might lead you to expect this action? (5) Write an equation for the reaction.

(*c*) (1) In what previous experiment was phosphorus triiodide formed and by what means? (2) How did it react with water? (3) Write an equation for the reaction.

EXPERIMENT 88. **Preparation of Arsenic.**

Reference: **470–471.**

DISCUSSION: Many elements which do not occur uncombined in nature may be prepared by heating a mixture of the oxide of the element with carbon. If the elementary substance formed in this way by the reduction of the oxide is volatile at the temperature of the reaction, it will pass off along with the oxide of carbon and be deposited again as a solid in the cold part of the apparatus.

MATERIALS: Arsenious oxide, powdered charcoal, lime water.

DIRECTIONS: Mix thoroughly about 1 gram of arsenious oxide with three times its volume of powdered wood charcoal and place the mixture in the bottom of a test tube, clamped to the ring-stand in a horizontal position, and fitted with a one-hole stopper and delivery tube, which dips into lime water in a test tube. Heat the mixture until a dark deposit is observed near the mouth of the tube and then at once remove the delivery tube from the lime water. (1) Why? (2) What is the deposit? (3) Was any carbon dioxide formed? (4) What is the evidence? (5) Write an equation for the reaction upon heating. (6) What kind of reaction did the arsenious oxide undergo? (7) What was the agent? (8) What process is illustrated by the appearance of the deposit at a distance from the place at which it was formed?

EXPERIMENT 89. **Comparison of Phosphorus, Arsenic, Antimony, and Bismuth.**

Reference: **449, 474, 478, 480, 485, 488, 490, 494, 495.**

DISCUSSION: Comparison of the compounds of elements in

the same group of the periodic classification shows that with increasing atomic weights their non-metallic or electro-negative properties decrease and their metallic or electro-positive properties increase. This relation is very clearly exemplified in Group V in the case of phosphorus, arsenic, antimony, and bismuth.

Positively charged ions of phosphorus are not known. Positive ions of arsenic presumably exist in small amounts in strongly acid solutions, since sulphides of arsenic can be precipitated by hydrogen sulphide. Upon passing down the series to bismuth, well-defined metallic salts are found to be its most stable compounds.

The hydroxides of phosphorus are entirely acidic, those of arsenic markedly so, whereas the hydroxide of antimony must be treated with an excess of a strong base to convert it into a salt, and the hydroxide of bismuth remains insoluble even in a large excess of a strong base.

The action of the chlorides of these elements still further illustrates the progressive change in their chemical properties. The complete hydrolysis of the chlorides of phosphorus has already been observed. Arsenious chloride becomes hydrolyzed to a very great extent although some arsenious ions still exist in solution, as was stated above. Antimony chloride forms an oxychloride and bismuth chloride a basic chloride when these compounds are added to water. These facts indicate partial hydrolysis in both cases.

The sulphides of arsenic and antimony are sufficiently acidic to form soluble compounds with ammonium sulphide; but bismuth is so electro-positive that its sulphide does not act in this way.

MATERIALS: Arsenious oxide, antimony trichloride, bismuth chloride, hydrogen sulphide, solutions of sodium hydroxide, concentrated hydrochloric acid, ammonium sulphide, litmus paper.

DIRECTIONS: (*a*) (1) In what experiment did you obtain a hydroxide of phosphorus? (2) Write an equation for the reaction by which it was formed.

To a pinch of arsenious oxide in a test tube add 5 c.c. of

water. (3) How does the vigor of the action compare with that of an oxide of phosphorus with water?

Add 5 c.c. of sodium hydroxide and boil the solution until it is clear. (4) Write an equation for the reaction taking place. (5) What is the name of the arsenic compound? (6) Of what acid is it a salt?

Add hydrochloric acid in excess, as shown by the action on litmus paper. (7) Write an equation to show the reaction of double decomposition.

Bubble hydrogen sulphide into the solution. (8) Describe and name the compound which appears. (9) What ion must have been present in the solution, in order that hydrogen sulphide could form the compound precipitated? (10) Write an equation to show how arsenious sulphide was formed.

Filter out the precipitate and wash it on the filter paper with hot water until the filtrate is only faintly acid. Rest the funnel in a clean test tube, poke a hole in the tip of the filter paper, and wash the precipitate through into the test tube with 5 c.c. of ammonium sulphide. (11) What has happened to the precipitate? (12) Write an equation for a hypothetical reaction between one molecule of arsenious oxide and three molecules of sodium oxide. (13) Write beneath this equation a similar one in which sulphur is substituted for oxygen and the ammonium radical for sodium. (14) For what reaction which you have carried out is this the equation? (15) What chemical characteristic of arsenic is the cause of this reaction?

Add an excess of hydrochloric acid to the solution in the test tube. (16) What appeared? (17) Write an equation for the reaction.

(*b*) Dissolve a small lump of antimony trichloride, about the size of a pea, in 5 c.c. of concentrated hydrochloric acid in a test tube. Add water to this solution, a few cubic centimeters at a time, until a precipitate appears. (1) What is the precipitate? (2) Write an equation to show how it was formed.

Add concentrated hydrochloric acid drop by drop until the precipitate disappears. (3) What relation does this reaction bear to the preceding one?

Bubble hydrogen sulphide into the solution. (4) Describe and name the compound formed. (5) Write an equation for the reaction.

Filter out the precipitate, wash it, and treat it with ammonium sulphide as you did under corresponding circumstances with the arsenic compound. (6) What happened? (7) Write an equation for the reaction.

Try the effect of acidifying the solution. (8) What appeared? (9) Write an equation for the reaction.

To 5 c.c. of a solution of antimony trichloride add very slowly sodium hydroxide solution until finally a considerable excess is present, as shown by litmus paper. (10) What changes did you observe? (11) Write equations for the reactions.

Repeat the last experiment with the substitution of ammonium hydroxide for sodium hydroxide. (12) What happened? (13) How do you account for the difference in behavior in these cases?

(*c*) Dissolve a small lump of bismuth chloride in 5 c.c. of concentrated hydrochloric acid and add water slowly as in the experiment with antimony chloride. (1) Record what happened. (2) Write an equation for the reaction. (3) What is the relation of the compound formed in this case to that formed with antimony chloride?

Add concentrated hydrochloric acid slowly until the precipitate disappears and then bubble in hydrogen sulphide. (4) Name and describe the compound precipitated. (5) Write an equation for its formation.

Filter and wash the precipitate and try the action of ammonium sulphide with it. (6) What happened?

To 5 c.c. of a solution of bismuth chloride add sodium hydroxide slowly and finally in excess. (7) What was formed? (8) How did the action differ from the corresponding experiment with antimony chloride? (9) Write an equation for the reaction.

(*d*) (1) In which group of the periodic classification do the elements studied in this experiment fall? (2) What other elements which you have studied are in this group? (3) Arrange the elements of this group in the order of their increasing

atomic weights. (4). Which of these five differs most widely from the others in its chemical properties? (5) In what other group have you found the element occupying the similar position differing from the other members? In making the following comparisons omit nitrogen from consideration. (6) What is the order of the elements of the fifth group in respect to their decreasing electro-negative nature? (7) What experiments show the relative positions of arsenic and phosphorus? (8) Of arsenic and antimony? (9) Of bismuth and antimony? (10) Formulate a test by which you could recognize a compound of arsenic. (11) A compound of antimony. (12) A compound of bismuth.

ORGANIC COMPOUNDS

EXPERIMENT 90. **Properties of Cellulose.**

References: **499, 502.**

DISCUSSION: Cellulose is produced in the growth of plants and is the chief constituent of wood and cotton. The purified material is used in making paper and cotton and linen cloth. Silk and wool are animal products, containing nitrogen, which are called proteins; wool also contains sulphur. The behavior of cellulose with acids and alkalies is quite different from that of proteins; as a consequence it is possible to distinguish by chemical means cotton from silk and wool. The presence of sulphur in wool serves to distinguish it from silk, which does not contain this element.

Cellulose is an alcohol, and like other compounds of this class it forms a nitrate when treated with strong nitric acid. A number of different nitrates of cellulose can be made in this way. The one containing the highest percentage of nitrogen is called gun-cotton, and is used in smokeless powder; the one containing a smaller percentage of nitrogen is used in making collodion and celluloid. In the experiment described below the conditions are given that yield the product which can be used to make colloidon.

MATERIALS: Pieces, about 1 inch square, of cotton, silk, and wool; raw cotton; concentrated hydrochloric and nitric acids; 10 per cent solution of sodium hydroxide; solution of lead acetate; alcohol; ether.

DIRECTIONS: (*a*) In separate test tubes shake pieces of cotton, silk, and wool with about 10 c.c. of cold concentrated hydrochloric acid, until one of them dissolves. (1) Record the result in each case.

(*b*) Place a piece of cotton and a piece of wool in a mixture of 1 c.c. of concentrated hydrochloric acid and 5 c.c. of water.

Press out the solution, and dry the pieces on the steam bath or set them aside until the next exercise. Try to tear the material. (1) Record the results.

(*c*) Place in a test tube a piece of cotton and one of wool and add 10 c.c. of a 10 per cent solution of sodium hydroxide. Heat the tube to such a temperature that it can just be held in the hand and shake for some time. (1) Record the result.

(*d*) Add a few drops of a solution of lead acetate to 20 c.c. of a 10 per cent solution of sodium hydroxide. Divide the solution into two portions. In one dissolve by heating a piece of wool and in the other a piece of silk. (1) Record and explain the results. (2) How can cotton, wool, and silk be distinguished from one another?

(*e*) Pour 10 c.c. of concentrated sulphuric acid into 10 c.c. of concentrated nitric acid. To the hot solution add 0.5 gram of raw cotton. At the end of 3 minutes withdraw the cotton, and drop it into a large amount of cold water. Wash the cotton in running water for about 1 minute, squeezing out the water from time to time. Set it aside to dry. Hold a small bit of the dry nitrate with tongs and place it in a flame. (1) Record the result. (2) What is the chemical composition of the substance?

Pour on to some of the nitrated cotton in a test tube 5 c.c. of a mixture of equal volumes of alcohol and ether. After a few minutes, pour off the clear liquid into a watch glass and let it evaporate slowly until all the liquid has disappeared. Place some water on the watch glass and after a few minutes lift the edge of the film and remove it from the glass. Dry it with filter paper. Hold a bit of the film with tongs and put it in a flame. (3) Record the result.

EXPERIMENT 91. **The Preparation of Soap.**

Reference: **502.**

DISCUSSION: The animal and vegetable fats are composed of the glycerine esters of acids of high molecular weight. When these esters are heated with a solution of sodium hydroxide they are converted into glycerine and the sodium salts of the acids, which are called soap. Soap is somewhat soluble in

water but is insoluble in a solution of sodium chloride. This fact is used in separating soap from water and glycerine, and from the excess of alkali used in the saponification of fats.

Materials: Lard, sodium hydroxide, toilet soap, sodium chloride, solution of calcium chloride.

Directions: (*a*) Weigh into a small beaker 20 grams of lard, and add to it a solution of 5 grams of sodium hydroxide dissolved in 5 c.c. of water. Rest the beaker in a second beaker containing boiling water and heat the mixture in this way for from 45 to 60 minutes. Stir the mixture occasionally until the contents hardens.

Remove the soap from the beaker. Add to a piece of it a drop of a solution of phenolphthalein. Test in the same way a piece of pure toilet soap. (1) Give a reason for the difference observed. (2) Write an equation for a reaction involved in the preparation of soap from lard, which contains the glycerine ester of stearic acid.

(*b*) Cut about one-half of the soap into thin shavings, place it in 150 c.c. of water, and gently boil the solution until the soap dissolves. Prepare 100 c.c. of a filtered saturated solution of sodium chloride and add it to the soap solution. Set the beaker away until the next exercise. (1) What was the action of the salt when it was added to the soap solution? (2) Why is this procedure used?

Remove the soap and let a part of it dry in a filter paper and use the rest for the following experiments. Dissolve a little of the soap in water and shake some of the solution in a test tube. (3) Record the result. Add calcium chloride to some of the solution. (4) What happened? Filter off the precipitate and shake some of it with water. (5) Is the precipitate soap? Give a reason for your answer. (6) What is the precipitate? Stearic acid is insoluble in water. (7) How could it be obtained from soap?

SILICON AND BORON

EXPERIMENT 92. **Silicic Acid and Silica.**

Reference: **508.**

DISCUSSION: Various silicic acids are known, which may be regarded as hydrates of silicon dioxide. Unlike the cases which have already been studied, these acids can not be prepared by the action of the anhydride on water, because silicic anhydride is extremely insoluble. They may be formed, however, by the double decomposition between a silicate and an acid, the particular silicic acid produced being dependent on the substances used, and on their concentration and temperature. The fully hydrated orthosilicic acid, H_4SiO_4, is a jelly, which can be completely dehydrated upon heating.

MATERIALS: Solution of sodium silicate, concentrated hydrochloric acid.

DIRECTIONS: (*a*) To 10 c.c. of sodium silicate in an evaporating dish add slowly with constant stirring 10 c.c. of dilute hydrochloric acid. (**1**) State how the substance formed appears and how it feels when rubbed between the fingers and thumb. (**2**) Assuming the original salt to be the meta silicate, Na_2SiO_3, write an equation for the reaction.

Evaporate the contents of the dish to dryness in the hood, and when the dish has cooled, thoroughly moisten the residue with concentrated hydrochloric acid. Again evaporate to dryness and then heat strongly for five minutes. When the dish is cool add 20 c.c. of water and stir to dissolve the sodium chloride. Carefully pour off the solution leaving the insoluble residue in the dish. (**3**) Describe its appearance and state how it feels when rubbed. (**4**) What is the substance? (**5**) Write an equation for the reaction by which it was formed.

Transfer the substance from the dish to a test tube and boil it with 10 c.c. of water. (**6**) What happened? (**7**) State

a striking difference between the chemical behavior of this compound and other acid anhydrides you have studied.

(*b*) (1) In what experiment did you study the action of an acid on silicon dioxide? (2) Write an equation for the reaction. (3) Do other acids act this way with silicon dioxide? (4) Upon what do you base your answer?

EXPERIMENT 93. **Compounds of Boron.**

Reference: **519–523.**

DISCUSSION: The comparative insolubility of boric acid in cold water makes it possible to prepare the acid from one of its soluble salts by double decomposition with another acid.

Borax, $Na_2B_4O_7$, may be regarded as a compound of one molecule of sodium oxide with two of boric anhydride, $Na_2O, 2B_2O_3$. Thus, enough boron exists in borax to form sodium meta borate, Na_2O, B_2O_3, and also to unite with a molecule of some other basic oxide to form another borate. When borax is fused with the oxide of a metal, this reaction takes place and the glassy mass of sodium meta borate becomes mixed with the borate of the other metal. Many of these borates possess characteristic colors, which may serve as tests for identifying the metals.

In making the special tests for compounds of boron it is necessary first to convert the compound to boric acid by adding an acid to the substance to be tested.

MATERIALS: Borax, alcohol, manganese dioxide, turmeric paper, concentrated hydrochloric acid, solutions of cobalt nitrate, and copper sulphate.

DIRECTIONS: (*a*) Dissolve 10 grams of borax in 15 c.c. of boiling water in a small beaker and add 5 c.c. of concentrated hydrochloric acid. Set the solution aside to cool and then filter out the crystals of boric acid which will have precipitated. (1) Describe the appearance of boric acid. (2) Write an equation to show how it was formed. (3) Why does the reaction take place?

(*b*) Put part of the crystals obtained in (*a*) into an evaporating dish, add 1 c.c. of concentrated hydrochloric acid, 10 c.c. of alcohol, and ignite the solution by directing the flame

of the burner for a moment into the dish. (1) What color is the flame? (2) What use may be made of this coloration of the alcohol flame?

(*c*) Dissolve about 1 gram of borax in a test tube half full of hot water, add 5 c.c. of dilute hydrochloric acid and wet a strip of turmeric paper with the solution. Dry the paper at 100°. This can be done conveniently by wrapping the wet paper around the upper part of a test tube containing about 5 c.c. of water and by then boiling the water until the paper is dry. (1) What color is the dried paper? Touch the paper with a drop of ammonium hydroxide. (2) What color is produced? (3) For what is this action on turmeric a test? (4) Why was the hydrochloric acid added?

(*d*) Make a loop about 3 mm. in diameter in the end of a platinum wire by winding it around the tip of a pencil. Alternately heat the wire in the Bunsen flame and dip it into powdered borax, until a clear glassy bead is formed in the loop of the wire. Touch the bead with a glass rod which has been dipped into a solution of cobalt nitrate, taking care that only a very small drop of the solution reaches the bead. Heat the bead in the flame until clear. (1) What color is it? (2) Write an equation to show what happens to cobalt nitrate when heated; (3) an equation for the reaction taking place between this compound and borax when fused. (4) Of what does the bead consist? (5) What gives it the color? After use, the bead may be removed by dipping it, while hot, into water. The sudden cooling shatters the bead, which may then easily be scraped from the wire.

Make another borax bead as before, touch it with a solution of copper sulphate, and fuse it in the oxidizing part of the flame. (6) What is the color of the bead? Heat the bead again in the reducing flame, that is, just at the tip of the blue inner cone. (7) What is the color of the bead?

Repeat the experiment, using a minute quantity of manganese dioxide instead of the copper sulphate, and heating in both parts of the flame. (8) What are the colors? (9) What use is made of the formation of these colored beads?

PHYSICAL PROPERTIES OF THE METALS. ALLOYS

EXPERIMENT 94. **Density of Metals.**

Reference: **528.**

DISCUSSION: It is easy to distinguish samples of the common metals from one another by a determination of their densities, because the values of the latter vary to such an extent that very accurate determinations are not necessary. The method described in Experiment 30 can be conveniently used. If the metals in the form of sheets are available, samples about 6 inches wide can be rolled so they will slip into a graduated cylinder. If sheet metal is not available any form of such a size that it can be placed in the cylinder can be used.

MATERIALS: Samples of several metals weighing from 50 to 75 grams each.

DIRECTIONS: Identify the samples of the metals furnished by a determination of the density of each in the way described in Experiment 30 *b*. (1) Tabulate your results giving in each case in separate columns the name of the metal, its weight, its volume, its density as found by you, and the recorded density.

EXPERIMENT 95. **The Hardness of Metals.**

Reference: **529.**

DISCUSSION: It is possible to discover which of two substances is the harder by determining which one scratches the other. Tests of the hardness of metals can be most readily made by attempting to scratch the surface of one metal with the sharp edge of a piece of the other metal in the form of a sheet.

MATERIALS: Pieces of sheet aluminium, copper, lead, tin, nickel, iron, zinc.

DIRECTIONS: Attempt to scratch the surface of one metal with the sharp edge of another. Place the harder to the left of the softer metal. Attempt to scratch the surface of each

of these two metals with a third metal and place it in its proper place, the harder metal to the left of the next softer. Continue with the rest of the metals. (1) Write down the names of the metals in the order of hardness found and place after each the hardness as given in the table on page 443 of the text.

EXPERIMENT 96. **Alloys of Lead and Tin.**

Reference: **538.**

DISCUSSION: The melting points of the alloys of lead and tin, which are used as solders, vary with the percentage of the two metals in the alloy. Pure tin melts at 232°. When the molten metal is allowed to cool, it behaves in a manner which is characteristic of other pure substances. The temperature falls rapidly until the freezing point, which is the same as the melting point, is reached. If the metal is not allowed to lose heat too rapidly, the temperature remains constant during solidification because, during the change in state, heat is given off at such a rate that it overcomes the cooling effect of the surrounding air. When the metal has solidified it again falls rapidly in temperature. By noting at equal intervals of time the temperature of the metal when it cools and changes from the liquid to the solid condition, it is possible to determine its freezing point. If the temperatures are plotted on a diagram in which time and temperatures are the coördinates, the curve obtained by connecting the points made for each observation will be found to have a section parallel to the axis of time. This temperature is the freezing point of the metal.

Pure lead, which melts at 327°, exhibits the same behavior as tin when it changes from the liquid to the solid state.

When a small amount of lead is added to tin, the molten mixture begins to solidify at a temperature below the freezing point of tin. As the amount of lead in the mixture is increased the point at which solidification begins continues to fall until the mixture is reached which contains 69 per cent of tin and 31 per cent of lead. As more lead is added the temperature at which solidification begins rises. The mixture of 69 per cent

of tin and 31 per cent of lead freezes at a definite temperature and in this respect resembles a pure compound; it is called the eutectic.

Other mixtures do not freeze at a definite temperature; as the molten alloy cools, the constituent which is in excess begins to separate at a definite temperature; the temperature continues to fall because the composition of the liquid changes as the result of the separation of the pure metal. When the composition of the liquid becomes that of the eutectic the temperature at which freezing takes place becomes constant, namely, that at which the eutectic freezes.

MATERIALS: Tin, lead, high boiling oil ("Nujol"), 360° thermometer.

DIRECTIONS: (*a*) In making determinations of the melting points of tin and its alloys with lead it is necessary to have enough of the metal to cover, when liquid, the bulb of the thermometer used. To determine this, place in a test tube just enough water to cover the bulb of the thermometer when it rests on the bottom of the tube. Measure the volume of the water. This number multiplied by 7.3 (the density of tin) will give the weight of tin required. (1) Weigh on a piece of paper on the platform scales approximately this amount of tin; the weighing should be to 0.1 gram. The pieces of the metal should be of such a size that they will readily slip into a test tube. Put into a perfectly dry test tube about 5 c.c. of a high boiling oil ("Nujol") and support the tube in a vertical position by means of a clamp and ring-stand at such a height that it can be heated by a burner.

Place a few pieces of tin in the oil and heat the latter until the tin melts. Drop into the heated tube pieces of the metal as rapidly as they melt. When all the metal has been melted, cautiously insert a perfectly dry thermometer into the liquid. Do this by first touching the surface of the liquid with the tip of the thermometer and removing the latter. Insert the thermometer again a little deeper and remove it immediately. The third time the thermometer can be left in the liquid. Rest the thermometer on the bottom of the test tube and heat the oil and metal until the thermometer registers about 250°.

Read and (2) record rapidly the temperature, count four slowly, record the temperature, count four, record, etc. Continue the readings in this way until the temperature has fallen to 150°. In recording the temperatures it is well to write on a piece of paper and to transfer the readings later to the note book. The eyes should be kept on the thermometer as the temperature falls, and the record made on the fifth second.

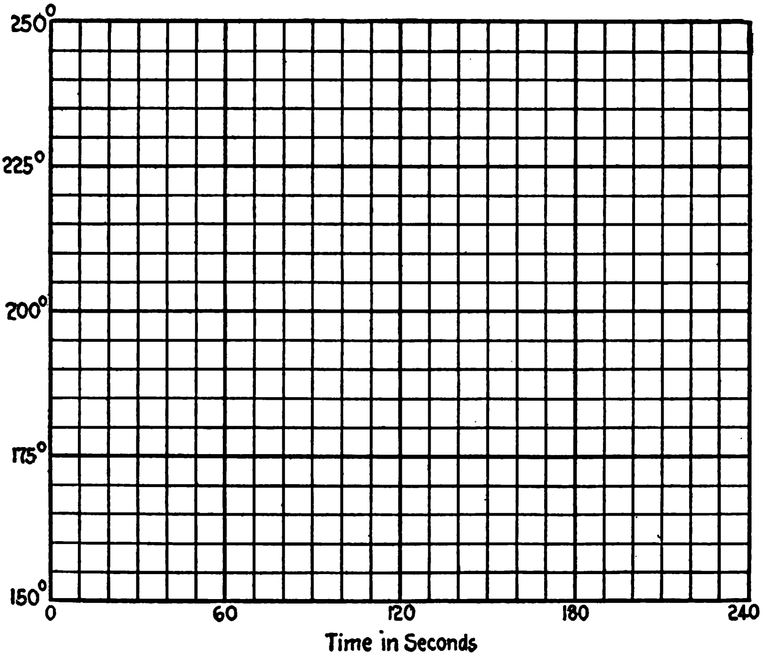

FIG. 15.

(3) Plot the temperatures against the time on Fig. 15, and draw a curve through the points.

(*b*) (1) Calculate what weight of lead must be added to the weighed amount of tin in the test tube to produce the eutectic, which contains 69 per cent tin and 31 per cent lead. (Lead to be added $= \frac{\text{wt. Tin}}{69} \times 31$). Weigh this amount to 0.1

gram. Heat the test tube containing the tin until the metal melts and add the lead slowly. Heat the tube to about 250° and (**2**) observe the temperatures at intervals of 5 seconds as it cools to 150°. (**3**) Plot the temperature on Fig. 15, and draw a curve through the points.

(**4**) Record the correct values of the melting point of tin and that of the eutectic, and those you obtained. Add to your observed value 6° in the case of the melting point of tin and 3° in the case of the eutectic. These values are the so-called "stem corrections," that is, the amounts that must be added to the observed temperatures to correct for the error due to the fact that the mercury in the stem of the thermometer was not heated to the temperature of the mercury in the bulb.

(*c*) Weigh out twice as much lead as the amount used in (*b*) above and add it slowly to the melted eutectic. When all the metal is liquid, heat the mixture to 250° and (**1**) record the temperature at intervals of 5 seconds as before until it falls to 150°. (**2**) Plot the temperatures and connect the point to make the so-called cooling curve. (**3**) Give a reason for the fact that the slant of the part of the curve to the melting point of the eutectic is different in this case from that of the other two curves. (**4**) Calculate the percentage composition of the alloy which gave this cooling curve. (**5**) At what temperature did solid begin to separate from the molten alloy? (**6**) Are the results in accord with the diagram in Section **538** of the text?

CHEMICAL PROPERTIES OF THE METALS

EXPERIMENT 97. **Behavior of Metals with Water.**

Reference: **545.**

DISCUSSION: The metals vary widely in their activity with water. Some, like sodium, decompose water rapidly at room temperature; others do not react even with steam at a high temperature. When a metal reacts with water, hydrogen and a hydroxide of the metal are formed. If these adhere firmly to the surface of the metal, reaction soon ceases, as they prevent contact between the metal and the water. If a metal is brought in contact with a second metal below it in the electromotive series, a so-called "couple" is formed. One of the reasons why a metal is more reactive when coupled with a second one is as follows: As the more active metal reacts, the hydrogen is evolved from the less active metal; it does not prevent, therefore, contact between the water and the active metal. A couple can be made by bringing into contact the two metals. It is usually made, however, by adding a solution of a salt of the less active metal to the more active metal. The latter liberates the former and thus its entire surface becomes coated with the less active metal. Couples made in this way are more active than those prepared by simple contact of the two metals, because larger areas of the two metals are in contact. Mercury or copper is commonly used as the less active metal in making couples.

MATERIALS: Magnesium ribbon; small piece of aluminium, tin, zinc, copper, and lead; solutions of mercuric chloride and of copper sulphate.

DIRECTIONS: In order to avoid mistaking for hydrogen the dissolved air liberated when the water used in the experiment is heated, boil in a beaker for about 1 minute about 250 c.c. of water. Pour about 10 c.c. of the boiled water into each of six test tubes and put them in the hot water in the beaker

which has been removed from the flame. Place a bit of magnesium ribbon and small pieces of aluminium, tin, zinc, copper, and lead in each of the tubes respectively. (**1**) Is hydrogen given off in any case?

Add to each tube, except the one containing zinc, 2 or 3 drops of a solution of mercuric chloride. To the zinc add 2 or 3 drops of a dilute solution of copper sulphate. (**2**) Which metals evolve hydrogen? (**3**) Arrange in a column the metals in the order of decreasing activity, as judged by the rate of evolution of gas. Place side by side with this arrangement the symbols of the metals in the order of their position in the electromotive series. (**4**) Is the order in the two arrangements the same? (**5**) Explain the lack of action with water alone. (**6**) Why did the addition of solutions of mercuric chloride and of copper sulphate activate the metals?

EXPERIMENT 98. **Action of Non-oxidizing Acids on Metals.**

References: **546, 547.**

DISCUSSION: Aqueous solutions of non-oxidizing acids react with metals down to hydrogen in the electromotive series. Whether the metal reacts completely or not is determined by the properties of the salt formed. If it is insoluble in water and adheres to the surface of the metal in such a way that the latter can not come into contact with the acid, the action soon ceases. The chlorides of all the metals above hydrogen in the electromotive series, except lead chloride, are freely soluble in water; all these metals except lead react in cold dilute hydrochloric acid. Lead chloride is soluble in hot water; as a consequence the metal reacts with hot hydrochloric acid.

In a similar way the reaction of metals in dilute sulphuric acid is determined by the solubility of the sulphates of the metals. Lead sulphate is insoluble in water but is soluble in concentrated sulphuric acid; the metal does not react with the dilute acid but does react with concentrated sulphuric acid.

The behavior of aluminium appears to be abnormal as it is only slightly attacked by sulphuric or nitric acids. This is probably due to the formation of a protective coating of insoluble basic sulphate or nitrate.

When a metal reacts with an acid to set free hydrogen, the salt formed is always that derived from the metal in its lower valence, since hydrogen is a reducing agent.

Materials: Lead, iron filings, concentrated hydrochloric acid, solutions of ferrous sulphate and of sodium carbonate.

Directions: (1) Make a table to indicate the behavior of the following metals with dilute hydrochloric acid and with dilute sulphuric acid: magnesium, aluminium, zinc, iron, tin, lead, and copper. If you are not sure of the action in any case, make the test. (2) In the cases where there is no action, state the reasons for the inactivity.

Place a bit of lead in a few cubic centimeters of concentrated sulphuric acid. (3) Does the metal react? (4) Why?

To determine which chloride of iron ($FeCl_2$ or $FeCl_3$) is formed when the metal dissolves in hydrochloric acid, add a few pieces of iron filings, or a small nail, to about 3 c.c. of concentrated hydrochloric acid. While the metal is reacting add a few drops of a solution of a ferrous salt to a solution of sodium carbonate. (5) Record the color of the precipitate. Repeat, using a ferric salt. (6) Record the color of the precipitate. Next pour a few drops of the solution prepared by the action of iron in hydrochloric acid, into a solution of sodium carbonate. (7) What is the color of the precipitate? (8) Is the compound formed from the metal and hydrochloric acid a ferrous or a ferric salt? (9) From what fact would you have predicted this result?

Experiment 99. **The Action of Oxidizing Acids on Metals.**

Reference: **548.**

Discussion: The behavior of a metal with an oxidizing acid is determined by the activity of the two substances involved. The higher the metal is in the electromotive series the more readily it is oxidized. The concentration of the acid is an important factor in its activity as an oxidizing agent. Since the ionization of the acid increases with dilution and since the undissociated acid alone brings about oxidation, the activity of the acid as an oxidizing agent increases as the concentration of the acid increases.

MATERIALS: Tin, aluminium, iron nail, concentrated nitric acid, solutions of stannous chloride, stannic chloride, ferric chloride, ferrous sulphate, ammonium sulphide.

DIRECTIONS: (*a*) Put a small piece of tin in a test tube and add to it dilute nitric acid made by mixing 1 c.c. of concentrated nitric acid with 10 c.c. of water. Allow the tube to stand for about one-half hour and meanwhile try the following test to distinguish a stannous from a stannic salt.

Add to a solution of stannous chloride, $SnCl_2$, a few drops of ammonium sulphide. (**1**) Record the color of the precipitate, which is stannous sulphide, SnS. Repeat with a solution of stannic chloride, $SnCl_4$. (**2**) Record the color of the precipitated stannic sulphide, SnS_2.

At the end of the half hour determine whether a stannous salt or a stannic salt is formed as the result of the action of dilute nitric acid on tin. (**3**) State what you did, the result, and the conclusion.

(*b*) Place about 1 gram of tin in a test tube and add 5 c.c. of concentrated nitric acid. After reaction has ceased, add water to the tube until it is full and shake. The precipitate is an oxide of tin. Filter about 5 c.c. of the liquid into another tube and add a few drops of ammonium sulphide to the filtrate. (**1**) Has any tin passed into solution? (**2**) What is the evidence?

Mix about 5 c.c. of the original milky liquid with an equal volume of a solution of sodium hydroxide and boil the mixture for about 1 minute. The oxide dissolves very slowly in a hot solution of sodium hydroxide, but it is not necessary to wait until it has all dissolved before proceding. Cool the test tube, add dilute hydrochloric acid until the solution is acid and then a few drops of ammonium sulphide. (**3**) Record the color of the precipitate. (**4**) What is it? (**5**) What is the valence of tin in this compound? (**6**) What do (*a*) and (*b*) in this experiment show in regard to the action of dilute and of concentrated nitric acid on tin?

(*c*) Place a little steel wool or an iron nail free from rust in a test tube and cover it with about 10 c.c. of a solution made by mixing 1 c.c. of concentrated nitric acid with 20 c.c. of water. Set the tube aside for about 10 minutes and then add

some of the solution to a little sodium carbonate solution. (1) Record the result. (2) Is a ferrous or a ferric salt formed? (3) What is the evidence?

(*d*) Treat a little iron with a mixture of one part of nitric acid and five of water. If the metal does not appear to react heat the tube gently. Add a little of the solution to a solution of sodium hydroxide. (1) What was formed? Make a statement as to the action of concentrated and moderately dilute nitric acid on iron.

(*e*) Heat a piece of aluminium with dilute and with concentrated nitric acid. (1) Is there evidence of any action in either case?

EXPERIMENT 100. **The Effect of the Presence of Oxygen on the Action of Acids on Metals.**

Reference: **549, 550.**

DISCUSSION: Metals that either do not displace hydrogen from acids or react with acids very slowly are more or less rapidly attacked by acids in the presence of oxygen. It is probable that the oxygen converts the metal at the surface into an oxide, which then dissolves in the acid.

MATERIALS: Strips of copper and of lead, dilute acetic acid.

DIRECTIONS: Place in a small beaker about 20 c.c. of dilute acetic acid and stand in the solution a strip of sheet copper so that a part of the metal is above the surface of the liquid. Arrange a similar experiment using lead instead of copper. Set the beakers in your desk and examine them at the next exercise. Test the two solutions to determine whether the metals have reacted by adding to each a few drops of ammonium sulphide. (1) What do you observe? (2) What does this prove? (3) Describe the appearance of the metals above and below the line at the surface of the liquid. (4) Give an explanation of the appearance.

EXPERIMENT 101. **The Action of Metals on Alkalies.**

Reference: **554.**

DISCUSSION: A number of metals form hydroxides that dissolve in both acids and alkalies. When the hydroxide dis-

solves in an acid it is playing the part of a base. When the hydroxide dissolves in an alkali it is playing the part of an acid. Most metals that form hydroxides which dissolve in alkalies are attacked more or less by alkalies. Aluminium, for example, reacts rapidly with a solution of sodium hydroxide, and the metal passes into solution as a salt which is called sodium aluminate. The same substance is formed when aluminium hydroxide dissolves in sodium hydroxide:

$$Al(OH)_3 + NaOH = NaAlO_2 + H_2O$$

Other metals of this class react very slowly with alkalies. Some react only when the metal is heated to a high temperature with the solid alkali.

The metals can be activated toward alkalies by coupling them with other metals, just as they are activated toward water and acids. When zinc is in contact with iron the former reacts rapidly with a warm solution of sodium hydroxide. Hydrogen is evolved from the iron and the zinc passes into solution as sodium zincate, Na_2ZnO_2. Tin behaves in a similar way and sodium stannite, Na_2SnO_2, is formed. It should be noted that in these cases the metal that dissolves is the one which forms a hydroxide soluble in sodium hydroxide. When two metals are coupled in neutral or acid solution hydrogen is evolved from the metal having the lower solution pressure—that is, the one lower in the electromotive series. When the solution contains an alkali, this is not always the case. Zinc is above and tin is below iron in the electromotive series, but when activated by iron in the presence of an alkali they each dissolve. The solubility of the hydroxide in alkalies is the determining factor in the reaction. The zinc that passes into solution is not presnt in the form of zinc ions, but as zincate ions, ZnO_2^{--} which result from the ionization of the salt formed.

MATERIALS: Aluminium; zinc; tin; solutions of zinc sulphate, ferrous sulphate, and ammonium sulphide.

DIRECTIONS: In separate test tubes add a small amount of the following metals to a solution of sodium hydroxide:

Aluminium, zinc, and tin. (1) What is the action in each case? Heat the solutions. Drop into the tubes containing zinc and tin a clean small iron nail. (2) From which metal is the hydrogen set free?

To determine which metal has dissolved proceed as follows: After the reaction has taken place for about two minutes pour off the liquid from the tube containing the zinc and add to it a few drops of a solution of ammonium sulphide. (3) What is the color of the precipitate? To determine whether the sulphide precipitated is that of iron or zinc add ammonium sulphide to solutions of ferrous sulphate and of zinc sulphate. (4) What is the color of the precipitate in each case? (5) Did the iron or the zinc dissolve? (6) As what ion does the dissolved metal exist?

Pour off the liquid from the tin, make the solution just acid with dilute hydrochloric acid and add a few drops of ammonium sulphide. (7) What is the color of the precipitate? (8) What is it? (9) How do you know? (10) Which metal dissolved? (11) Why did it dissolve?

ELECTROCHEMISTRY

EXPERIMENT 102. **The Production of an Electric Current from Chemical Energy.**

Reference: **566.**

DISCUSSION: When zinc is placed in a solution of hydrochloric acid the metal dissolves, hydrogen is set free, and heat is developed. When copper is placed in dilute hydrochloric acid no reaction takes place. If zinc that is dissolving in hydrochloric acid is brought into contact with copper, the metal continues to pass into solution, and hydrogen is evolved from the copper. A part of the liberated energy that appeared as heat in the first case appears as electrical energy when the metals are in contact.

The explanation of the reactions in the two cases, using the conception of electrons is as follows: When zinc dissolves in an acid the atoms that pass into solution give up their electrons to the hydrogen ions and hydrogen is given off:

$$Zn + 2H^{+} = Zn^{++} + H_2 + \text{Heat}$$

When zinc is in contact with copper and the former passes into solution, the electrons that were on the zinc atoms before solution occurred pass from the zinc to the copper. It is from the latter metal that the hydrogen ions take up the electrons necessary to convert them into hydrogen gas.

In the experiment described below two cells made of zinc, copper, and sulphuric acid are set up and joined in series, that is, the copper of one cell is joined to the zinc of the next. In this way it is possible to obtain a current that will decompose solutions of acids, bases, and salts.

MATERIALS: Two copper and two zinc electrodes of the dimensions represented in Fig. 16. Four pinch cocks. A

solution of mercuric chloride containing 1 gram of the salt to 100 c.c. of water, of potassium iodide, sodium chloride, phenolphthalein, litmus paper.

Directions: Bend over to make a right angle the narrow end of one zinc and one copper electrode; slide a pinch cock over the end of one electrode, slip the other over the first, and release the pinch cock. Place one electrode in a small beaker and the second in another (see Fig. 16); place the second zinc electrode in a solution of 1 gram of mercuric chloride in 100 c.c. of water and let it stand for 3 minutes. Mercury is deposited evenly over the surface of the zinc and as a result the metal reacts very slowly with dilute acids. This procedure is used when zinc is employed in batteries to prevent the metal from dissolving as the result of the setting up of couples between the zinc and the impurities in it. By the "amalgamation" of the zinc a uniform amalgam of zinc and mercury is formed (**567**).

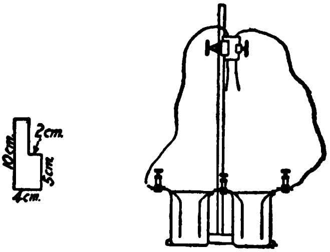

Fig. 16.

Place the second copper electrode in the beaker containing the zinc, and the zinc electrode in the beaker containing the copper. Join to each of these electrodes by means of pinch cocks a copper wire which passes through a cork as indicated in the figure.

(*a*) Add 10 c.c. of concentrated sulphuric acid to 200 c.c. of water, cool the solution, pour about 20 c.c. of it into a small beaker and the rest into the two beakers of the apparatus. (**1**) Record the action of the acid on the four electrodes.

Bring together the two terminals that pass through the

cork. (**2**) What happens on the electrodes? Separate the two terminals and hold the small beaker containing the sulphuric acid so that they dip under the surface of the liquid. (**3**) What occurs at the terminals? (**4**) Is the terminal from which the gas is given off connected with zinc or copper? (**5**) What is the gas? (**6**) Why is gas not given off from the other electrode? (**7**) What is the terminal called from which the gas is given off? (**8**) State the direction in which the electrons in the circuit are flowing. (**9**) In which way is the positive current flowing?

(*b*) Place a few cubic centimeters of a solution of potassium iodide in a small beaker and place the latter so the terminals dip into the solution. (**1**) State what happens at the two electrodes.

(*c*) Repeat the experiment using a solution of sodium chloride to which has been added a few drops of a solution of phenolphthalein. (**1**) State what is observed, and explain the result. (**2**) How could you determine which of the terminals of an electric circuit is the positive pole?

(*d*) Moisten a piece of litmus paper with a solution of sodium chloride, and press the paper against the two terminals. (**1**) Explain what happens.

(*e*) Cut off about 1 inch from the anode terminal and wind around it a piece of platinum wire. Leave the end of the wire free to use as the electrode. Dip the terminals into a solution of sulphuric acid. (**1**) Is oxygen evolved? (**2**) Why in this case and not when copper was used as the electrode?

EXPERIMENT 103. **Metallic Couples.**

Reference: **567.**

DISCUSSION: The activation of metals toward water by means of couples was illustrated in Experiment 97. The student should reread the discussion of that experiment in the light of the experiment on the production of an electric current (Experiment 102). In the following experiment additional couples are studied.

MATERIALS: Nail, strip of zinc, tin, lead, copper and aluminium.

Directions: (*a*) Wrap tightly a small piece of zinc around the head and another piece around the point of a wire nail free from rust. Attach two pieces of tin in a similar way to a nail. Place the nails side by side, but not touching each other, on the bottom of a beaker. Cover the nails with a layer of water about 3 cm. deep, and set them aside. At the next exercise examine the two nails, the zinc, and tin carefully. (1) Record the appearance of the iron in the two cases. (2) Give an explanation of what happened. (3) Is the appearance of the zinc and the tin in accord with the explanation? (4) What is the deposit on the zinc? (5) What practical conclusion can be drawn from the experiment?

(*b*) Devise and carry out an experiment to determine if contact with (1) lead, (2) copper, or (3) aluminium affects the rusting of iron. (1) Record the results in the three cases.

Experiment 104. **Faraday's Law.**

Reference: **569.**

Discussion: Faraday's law states (1) that the quantity of any one substance decomposed by the electric current is proportional to the quantity of electricity passed through its solution, and (2) that the quantities of two or more substances liberated by equal quantities of electricity are proportional to the chemical equivalents of these substances. The first part of the law can be tested by passing a constant current through a solution of a salt, such as copper sulphate, and determining the weight of copper deposited during different time intervals. When a constant current flows the quantity of electricity that passes is proportional to the time during which the current flows.

The second part of the law can be tested in the manner outlined in the following experiment. The direct current which is ordinarily furnished is one at 110 to 115 volts. By placing in the circuit a 60-watt lamp the current is reduced, as the result of the resistance of the lamp, so that it serves to deposit copper evenly on an electrode. In order to test Faraday's law such a current is passed first through a solution of copper sulphate in which are placed copper electrodes and then

through a solution of tin chloride, $SnCl_2$, in which are placed tin electrodes. As the current passes through the copper sulphate solution, the metal dissolves at one electrode and is deposited at the other. An analogous change takes place in the case of tin. The amounts deposited in the two cases should be in the proportion of the atomic weights of the two metals, because in the solutions used the metals have the same valence. On account of the fact that it is difficult to deposit tin in a form that adheres to the electrode, and can, as a consequence, be easily weighed, the amount of tin that dissolves is determined by noting the change in weight of the electrode from which the metal is dissolved. If pure tin is used this loss is equal to the gain in weight of the other electrode.

It should be remembered (**563**) in answering the questions given below that watts = volts × amperes, and that coulombs × seconds = amperes.

MATERIALS: Two electrodes made of thin sheet tin of the size indicated in Fig. 16. Two electrodes made of thin sheet copper of the same size. Copper wire. Four pinch cocks. Solutions of copper sulphate and of stannous chloride, each containing approximately 20 grams of the salt to 100 c.c. of water.

DIRECTIONS: Bend the smaller end of one of the copper electrodes over the edge of a small beaker, so that the electrode will hang in the beaker. The end outside the beaker should be bent into a horizontal position. Arrange the other copper electrode opposite the first. Place the two tin electrodes in a second beaker in the same way. Connect a 60-watt lamp with the source of electricity and with a copper wire which is joined to one of the copper electrodes in the following manner: Slip a pinch cock over the end of the wire, and rest the latter on the horizontal part of the electrode outside the beaker; open the pinch cock, and move it until it is over the joint and then close it. By means of a short piece of wire and two pinch cocks connect the second copper electrode with one of the tin electrodes. Connect the second tin electrode with the second terminal from which the current is taken. Remove the electrodes and scratch upon them with the point of a file the

numbers 1, 2, 3, and 4 respectively. Weigh each electrode to centigrams. Assemble the apparatus as before and cover the copper electrodes with the solution of copper sulphate described under "Materials," above, and the tin electrodes with the solution of stannous chloride. Switch on the current and note the time. Observe closely what happens in the two beakers. The solution of copper sulphate should be stirred gently from time to time. If this is not done all the copper in the part of the solution touching the electrode on which the metal is depositing will pass out of solution and hydrogen will be evolved by the current.

As the tin is deposited the crystals will grow out into the solution. Care should be taken to prevent contact between them and the other electrode. If the crystals grow out very far into the solution gently press them back to the electrode from which they have grown. Be careful not to break them away from the electrode.

At the end of from 45 minutes to 1 hour, break the current and note the time. Disconnect the electrodes. Hold the copper electrodes under running water for a few seconds and then dry them by pressing them against a clean towel. Wash and dry in the same way the tin electrode from which the tin was dissolved. Hang the tin electrode to which the crystals are attached in a beaker of water for a few minutes; remove the electrode and hang it a second time in water. Remove it and hang it on an empty beaker until it is dry. Weigh the four electrodes to centigrams.

(1) Tabulate on the opposite page the original weights of the electrodes, their weights after the metals have been deposited, and the gain or loss in weight of each electrode. (2) Is the weight of the copper dissolved equal to the weight of the metal deposited? (3) If not, give a reason for the difference? (4) Answer the same questions in regard to the tin. (5) Which is probably the more accurate measure in the two cases of the current that passed through the solutions? (6) Calculate from the weight of the copper deposited what weight of tin should have been deposited according to Faraday's law. Write under this the weight found in the experiment. (7)

Calculate from the weight of copper dissolved the weight of tin that should have been dissolved. Write under this the weight found in the experiment. (8) The weight of copper deposited should be the most accurate of the measurements made. Is the weight of the tin deposited or dissolved more in accord with the amount required by Faraday's law? (9) Calculate from the fact that a 60-watt lamp was placed in the circuit the current (amperes) that were used in the experiment. (The resistance of the solution is so small that it can be neglected.) (10) Calculate from the weight of copper deposited the number of coulombs that were used. (11) From this last result and the time required for the deposition, calculate the current in amperes. (12) Compare the result in (11) with that obtained in (9) above, and state which is the more accurate measurement.

EXPERIMENT 105. **The Lead Storage Battery.**

Reference: **578.**

DISCUSSION: If an electric current is passed through a solution of sulphuric acid between lead electrodes, hydrogen is evolved at one electrode and on the other a deposit of lead dioxide is formed. If the circuit is broken and the electrodes are connected by a wire, a positive current flows through the wire from the electrode on which the lead dioxide was deposited. The dioxide is converted into lead sulphate. At the other electrode metallic lead is also converted into lead sulphate. The equation for the reactions involved is as follows: $PbO_2 + 2H_2SO_4 + Pb = PbSO_4 + 2H_2O + PbSO_4 + \text{electricity}$. If next a current from an outside source is passed between the electrodes, reactions take place which are the reverse of those just given; the equation for the reaction is that given above when read from right to left. To force the reaction in this direction energy is necessary and this is supplied by the current. When the electrodes are joined the reaction reverses itself and energy in the form of electricity is set free. The equation read from left to right represents the changes involved when a lead storage battery is furnishing a current; read from right to left it represents the changes involved when the battery is being charged.

Materials: Two lead electrodes of the form and size described in Experiment 102, 2 pinch cocks, copper wire, voltmeter.

Directions: Place the electrodes in a small beaker containing dilute sulphuric acid made by adding 10 c.c. of concentrated sulphuric acid to 90 c.c. of water. Connect the electrodes by means of pinch cocks with two copper wires that are joined to a 110-volt circuit through a 60-watt lamp. Put on the current and observe at once what happens. Break the circuit and hold in contact with the two electrodes a piece of copper wire for about 5 seconds. Remove the wire, put on the current for about 5 seconds; break it, and connect the electrodes as before. Repeat charging and discharging in this way about 5 times.

Examine the charged electrodes and (1) describe their appearance. Test the voltage of the charged cell by means of a voltmeter such as those used in testing dry cells. (2) What voltage do you find? (3) What is the voltage of the lead storage battery? Charge the cell, remove the electrodes and pour on the surface of each a few drops of dilute hydrochloric acid and at once note the odor. (4) What do you detect? (5) From which electrode was the gas given off? (6) Write an equation for the reaction. (7) Write an equation for the reaction taking place when the cell is being charged.

PROPERTIES OF HYDROXIDES AND SALTS

EXPERIMENT 106. **The Effect of Ammonium Salts on the Precipitation of the Hydroxides of the Bivalent Metals by Ammonium Hydroxide.**

Reference: **589.**

DISCUSSION: The hydroxides of most of the bivalent metals which are precipitated by a solution of ammonium hydroxide are not precipitated by this reagent if an ammonium salt is present in the solution. The extent of the ionization of ammonium hydroxide is small and when an ammonium salt is added to the solution the ionization of the hydroxide is reduced as the result of the presence of the added ammonium ions. Such an addition forces the equilibrium represented by the equation

$$NH_4OH \leftrightarrows NH_4^+ + OH^-$$

in the direction which results in the formation of undissociated ammonium hydroxide. As a consequence, the concentration of the hydroxyl ions is reduced to such an extent that the solution no longer causes the precipitation of the hydroxides of the bivalent metals, on account of the fact that the latter compounds are slightly soluble in water.

The solubility of hydroxides of the trivalent metals is exceedingly small; as a consequence the hydroxides of the trivalent elements are precipitated by ammonium hydroxide in the presence of ammonium salts.

MATERIALS: Solutions of calcium chloride, magnesium chloride, ammonium chloride.

DIRECTIONS: (*a*) In a test tube add ammonium hydroxide to a solution of calcium chloride. Add a solution of sodium hydroxide to one of calcium chloride. (**1**) What happened in each case? (**2**) What is thus proved about the relative ioniza-

tion of ammonium hydroxide and of sodium hydroxide? (3) How did you arrive at this conclusion?

(*b*) Repeat (*a*) above using magnesium chloride instead of calcium chloride. (1) What happened? (2) Is the hydroxide of calcium or of magnesium the more soluble? (3) What is the evidence?

Add a solution of ammonium chloride to the tube to which ammonium hydroxide was added. (4) What occurred?

Add to a solution of magnesium chloride an equal volume of ammonium chloride solution and then ammonium hydroxide. (5) What was the result? (6) What is the effect of ammonium chloride upon ammonium hydroxide? (7) What is the reason for the result in this experiment? (8) What is the reason for (4)?

(*c*) Fit a filter paper in a funnel ready for use. Add to a solution of ferrous sulphate an equal volume of a solution of ammonium chloride. Add ammonium hydroxide to the mixture and filter at once. Ferrous salts are oxidized in solution by the air; as a consequence it is necessary to filter off the ferric hydroxide precipitated. (1) What is the appearance of the filtrate as it drops from the funnel? Shake the filtrate. (2) What occurred? The ferrous salt is oxidized by the air and the ammonium hydroxide present causes the precipitation of ferric hydroxide. (3) Will ammonium chloride prevent the precipitation by ammonium hydroxide of ferrous hydroxide? (4) Of ferric hydroxide? (5) What is the evidence?

EXPERIMENT 107. **Isomorphism of Salts.**

Reference: **591.**

DISCUSSION: When two salts that do not react with each other are allowed to crystallize from the same solution, crystals of the two are usually deposited side by side. If, however, the two salts resemble each other closely in crystalline form they form mixed crystals, the composition of which is determined by the proportions of the two salts present in the solution. When two salts form such mixed crystals, they are said to be isomorphous.

Ordinary alum has the composition represented by the

formula $K_2SO_4,Al_2(SO_4)_3,24H_2O$. Chrome alum has a similar formula, but contains chromium instead of aluminium, $K_2SO_4,Cr_2(SO_4)_3,24H_2O$. Both salts crystallize in octahedra. The aluminium salt is colorless and the chromium salt is purple. When mixtures of the two are allowed to crystallize together uniform mixed crystals are obtained, the color of which is determined by the proportions of the two salts in the solution.

MATERIALS: Potassium aluminium alum, potassium chromium alum.

DIRECTIONS: (*a*) Dissolve 16 grams of potassium aluminium alum in 100 c.c. of hot water, filter if necessary, and cool the solution to room temperature in running water. Shake occasionally for five minutes 8 grams of finely powdered potassium chromium alum with 100 c.c. of water at about 30°. The temperature should not be allowed to go higher than 35° in order to prevent the change of the chromium salt to the green modification. Filter the solution. In four beakers place the following: (1) 40 c.c. of the solution of aluminium alum and 10 c.c. of the chromium alum, (2) 20 c.c. of the former and 10 c.c. of the latter, (3) the rest of the solution of the former and (4) the rest of the solution of the latter.

(*b*) Set aside the solutions and examine them at the next exercise. Remove some of the crystals from each of the beakers and place them side by side on a piece of filter paper. **(1)** Are the crystals from each beaker uniform in appearance? Examine closely the crystalline form of the crystals. **(2)** How many faces are there on a single crystal? **(3)** State what form they have. **(4)** Compare the colors of the crystals. **(5)** What inference can you draw from the colors? **(6)** What is meant by isomorphous salts? **(7)** Can isomorphous salts be separated from each other by crystallization?

EXPERIMENT 108. **The Heat of Solution of Salts.**

Reference: **592.**

DISCUSSION: When a substance is dissolved in water there is a change in temperature of the solvent. The number of calories set free or absorbed when 1 gram-molecular-weight of a salt dissolves in such an amount of water that further addi-

tion of the solvent produces no heat change, is called the molar heat of solution of the salt. In the case of most salts the heat of solution is negative, that is, heat is absorbed and the temperature falls. The molar heats of solutions of salts vary widely. The experiment described below is designed to illustrate the variation in this quantity.

MATERIALS: Potassium nitrate, sodium chloride.

DIRECTIONS: (*a*) Grind in a mortar to a fine powder about 11 grams of potassium nitrate. (**1**) Weigh on a piece of paper on the platform scales 10.1 grams of the powdered salt. Measure into a 500 c.c. beaker 250 c.c. of water at room temperature and (**2**) read the temperature of the water to 0.1 degree. Pour the potassium nitrate into the water and stir with the thermometer. The salt should dissolve in less than 30 seconds. (**3**) Read the temperature when the salt has dissolved. (**4**) From the fall in temperature and the volume of the solution calculate the number of calories absorbed. Since the fall in temperature is small, the error due to the absorption of heat by the beaker and thermometer can be neglected. (**5**) What is meant by the molar heat of solution of a salt? (**6**) Calculate the molar heat of solution of potassium nitrate.

(*b*) Repeat the experiment described in (*a*) above replacing potassium nitrate by sodium chloride. Use 5.8 grams of the salt and 250 c.c. of water. (**1**) Record the results. (**2**) Calculate the molar heat of solution of sodium chloride.

EXPERIMENT 109. **Effect of Temperature on the Solubility of Salts.**

Reference: **592.**

DISCUSSION: The solubility of most substances increases with rise in temperature, but the rate of change varies greatly with different substances. In general, if the heat of solution of a salt is negative, that is, if the temperature falls when it dissolves in water, the salt is more soluble in hot than in cold water. Further, the change in solubility with change in temperature is greater in the case of a salt with a high heat of solution than in the case of one which produced a small heat effect when dissolved. In the previous experiment (number

108) it was found that there was a marked difference between the molar heats of solution of potassium nitrate and sodium chloride. The following experiment serves to illustrate the relation between the heat of solution of a salt and the change in the solubility of the salt with rise in temperature.

MATERIALS: Potassium nitrate, sodium chloride.

DIRECTIONS: (*a*) Grind to a powder 35 grams of potassium nitrate. Weigh the salt to decigrams on a piece of paper on the platform scales. Measure into a small flask or beaker 35 c.c. of water and set it aside. Fit a stopper to a 100 c.c. graduated cylinder so that when the latter is shaken the water will not leak out. Dry the graduate carefully and pour into it the potassium nitrate. Tap the cylinder on a book until the upper surface of the salt is level, and read the volume of the salt to 0.1 c.c. Tap the cylinder smartly again and read the volume again. Repeat the tapping until the volume remains constant. (1) Record the volume of the potassium nitrate.

Add to the graduate the 35 c.c. of water previously measured, insert the stopper, and shake vigorously for one-half minute. Let the salt settle, and read its volume. Shake again for about one-half minute and read the volume a second time. (2) Record the volume of the salt and the temperature of the solution. (3) What is the volume of the potassium nitrate that dissolved in the 35 c.c. of water used? (4) From the original volume of the dry salt and its weight calculate the weight of 1 c.c. of it. (5) From this value and the volume of the salt that dissolved calculate the weight of potassium nitrate that dissolved in the 35 c.c. of water at the temperature used. (6) What would dissolve in 100 c.c. of water?

(*b*) Place the cylinder in a beaker of water and heat until the temperature of the solution in the graduate is about 40°. Remove the graduate and shake it vigorously for one-half minute, let the salt settle, (1) measure its volume and immediately observe and record the temperature of the solution. (2) Calculate as before the solubility of potassium nitrate in 100 c.c. of water at the recorded temperature.

(*c*) Replace the graduate in the hot water and heat it until

the temperature is about 55°. Proceed as before, (**1**) recording the volume of the salt and the temperature, and (**2**) calculating the solubility.

(*d*) Plot the solubilities against temperatures in the way given in the diagram in **592** of the text. Mark also on your plot the correct values for the solubilities at these temperatures as obtained by inspection of the diagram in the text.

(*e*) In the way described above determine the solubility of sodium chloride at room temperature and at about 55°. Use in this case 25 grams of salt. Plot the curve as before and also the values obtained from the diagram. (**1**) What relation exists between the relative heats of solution of potassium nitrate and sodium chloride, as determined in Experiment 108 and the relative effects of rise in temperature upon solubility as found in this experiment?

EXPERIMENT 110. **The Solubility of Salts in Acids.**

Reference: **593.**

DISCUSSION: When an insoluble salt is treated with an acid, double decomposition may or may not take place depending upon the solubilities of the original salt and of that which may be formed, and upon the relative degrees of ionization and of solubility of the acid added and of the acid produced. For example, if the salt AB is treated with the acid HD, the reaction expressed by the equation

$$AB + HD \rightarrow AD + HB$$

may or may not take place, depending upon the solubilities of the salts AB and AD and upon the ionization and solubilities of the acids HD and HB. The properties of all four substances are involved.

Or viewed wholly from the ionic standpoint, if the concentrations of ions of B, depending upon the solubility of AB, and of ions of D, depending upon the degree of ionization of HD, are great enough to cause the continued formation of HB, the double decomposition will take place.

In the cases studied in this experiment, the salts represented by AD are to be highly soluble. The effect of variation in the

solubility of the salt *AB*, the effect of the ionization of the acid *HD*, and the effect of the solubility of the acid *HB* will be exemplified.

The action of a highly ionized acid on the salt of a polybasic acid, with the formation of a hydrogen salt of the latter, depends upon the same general principle as that stated above, although the case may appear slightly more complex. The action of nitric acid upon calcium phosphate serves as an illustration.

Calcium phosphate is slightly soluble in water and dissociates into its ions:

$$(1)\quad Ca_3(PO_4)_2 \rightleftarrows 3Ca^{++} + 2PO_4^{---}$$

The above equation indicates an equilibrium that exists between the ions of the dissolved salt and the undissolved solid.

When nitric acid is added, the ions of the acid are introduced into the solution.

$$(2)\quad HNO_3 \rightleftarrows H^{+} + NO_3^{-}$$

$$(3)\quad 2H^{+} + PO_4^{---} \rightleftarrows H_2PO_4^{-}$$

The equilibrium in equation (1) above is disturbed as the result of the removal of PO_4^{---} ions. As a consequence more of the solid passes into solution and finally it all dissolves.

The solution now contains the following ions: Ca^{++}, H^{+}, $H_2PO_4^{-}$, NO_3^{-}. If a base is now added to the solution, the H^{+} and the $H_2PO_4^{-}$ ions disappear as the result of neutralization. The $H_2PO_4^{-}$ ion reacts with the base as follows:

$$2Na^{+} + 2OH^{-} + H_2PO_4^{-} = 2Na^{+} + 2H_2O + PO_4^{---}$$

The calcium ions and the PO_4^{---} ions unite and $Ca_3(PO_4)_2$ precipitates.

This explanation makes it evident why bases precipitate from the solutions in acids of slightly soluble salts, the salt itself and not the hydroxide of the element.

MATERIALS: Solutions of zinc chloride, copper sulphate, ammonium sulphide, sodium carbonate, and acetic acid.

DIRECTIONS: (*a*) Prepare in a test tube some zinc sulphide by diluting 5 c.c. of a solution of zinc chloride with 5 c.c. of water and adding 5 c.c. of ammonium sulphide. Pour half the suspended precipitate into another test tube. Add dilute hydrochloric acid to one tube and acetic acid to the other. (1) What happened in each case? (2) Upon what difference in the two acids used does the difference in their action depend?

(*b*) Prepare some copper sulphide from copper sulphate in the same way you made zinc sulphide. Try the action of hydrochloric acid upon it. (1) What happened? (2) Why did the result differ from that with zinc sulphide and hydrochloric acid?

(*c*) Prepare some zinc carbonate from zinc chloride by adding sodium carbonate to the solution. Treat the zinc carbonate with acetic acid. (1) What happened? (2) What is the reason for the difference between this result and that obtained when zinc sulphide was treated with acetic acid in part *a*?

(*d*) Add a solution of sodium phosphate to a solution of calcium chloride. (1) Write an equation for the reaction. Add to the solution dilute nitric acid until the precipitate dissolves. (2) Write an equation for the reaction using ionic symbols.

Add sodium hydroxide to the solution until precipitation is complete. Determine as follows whether the precipitate is calcium hydroxide or calcium phosphate. Filter the solution, wash the precipitate with water until the filtrate no longer gives a test for a phosphate. Dissolve a part of the precipitate in dilute nitric acid and test for a phosphate. (3) How did you test for a phosphate? (4) What was the result with the nitric acid solution of the precipitate? (5) What was the precipitate?

EXPERIMENT 111. **The Solubility of Salts in a Solution of Ammonia.**

Reference: **595.**

DISCUSSION: The hydroxides and insoluble salts of certain metals dissolve in a solution of ammonia as the result of the

formation of soluble compounds which yield complex ions of the metal. Copper hydroxide, $Cu(OH)_2$, for example, is insoluble in water; it dissolves in a solution of ammonia as the result of the formation of a soluble compound which has the composition $Cu(NH_3)_4(OH)_2$. The ion $Cu(NH_3)_4{}^{++}$ gives a deep blue color to the solution. Similarly silver chloride forms the soluble compound $Ag(NH_3)_2Cl$.

MATERIALS: Solutions of copper and of zinc sulphates, of silver and of nickel nitrates.

DIRECTIONS: Add a few drops of sodium hydroxide in separate test tubes to solutions of a copper, silver, nickel, and zinc salt. (1) Write equations for the reactions that take place.

Add a solution of ammonia to each of the tubes. (2) Record the results. (3) Write equations for the reactions.

EXPERIMENT 112. **Preparation of a Double Salt: Ferrous Ammonium Sulphate.**

Reference: **598, 750.**

DISCUSSION: The so-called double salts are compounds which are formed as the result of the chemical combination of two salts. They are usually prepared by dissolving the two constituents in water and allowing the crystals to deposit from the solution. The composition of a double salt is definite; it does not vary with the proportions of the constituents present in the solution as is the case with the composition of the crystals obtained when mixtures of isomorphous substances are crystallized (see Experiment 107).

Double salts break down more or less completely into their constituents when dissolved in water. The presence of the separate ions of the constituents of the salt can be shown by appropriate tests.

MATERIALS: Ferrous sulphate, ammonium sulphate, solution of sodium carbonate, materials needed to test for an ammonium salt and for a sulphate.

DIRECTIONS: Add 28 grams of crystalline ferrous sulphate, $FeSO_4,7H_2O$ to 20 c.c. of water; heat just to boiling and filter hot if necessary. Add 13 grams of ammonium sulphate to 18 c.c. of water; heat and filter the hot solution if necessary.

Mix the two solutions and add 5 drops of concentrated sulphuric acid. If a salt crystallizes out, warm the solution until it dissolves. Cover the beaker containing the solution with a piece of filter paper and set it aside until the next exercise. Filter off the crystals and spread them on a paper to dry. (1) Describe the crystals. (2) What are they called? (3) To what class of substances do they belong? (4) Are they a mixture or a pure substance? (5) How could this be proved?

Dissolve a few of the crystals in water and test separate portions of the solution for a sulphate and for an ammonium salt. (6) State how the test for a sulphate was made and the result obtained. (7) State how the test for an ammonium salt was made and the result obtained.

Add to a solution of sodium carbonate a few drops of a solution of ferrous sulphate. (8) Record the result. Add to a solution of sodium carbonate a few drops of the solution of the double salt. (9) Record the result. (10) Does the latter give the test for a ferrous salt? (11) What conclusion can you draw as to the behavior of double salts when they are dissolved in water?

EXPERIMENT 113. **Properties of Complex Salts.**

Reference: **598.**

DISCUSSION: Complex salts are formed as the result of the union of two salts. They differ from double salts in that they do not break down in solution into the ions of the salts from which they are formed. Potassium ferrocyanide is an example of a complex salt. It can be prepared by dissolving ferrous cyanide in a solution of potassium cyanide:

$$Fe(CN)_2 + 4KCN = K_4Fe(CN)_6$$

In Experiment 112 it was shown that the double salt formed from ammonium sulphate and ferrous sulphate broke down in solution into its constituents; the solution gave the test for a ferrous salt. In the experiment described below the complex cyanide of iron and potassium is studied.

MATERIALS: Potassium ferrocyanide, solutions of ferrous sulphate and of sodium carbonate.

Directions: Dissolve a small bit of a crystal of potassium ferrocyanide in water and add a few drops of the solution to a solution of sodium carbonate. (1) Is a precipitate formed? Add a few drops of a solution of ferrous sulphate to a solution of sodium carbonate. (2) What is the appearance of the precipitate? (3) Does the solution of the complex salt show the presence of a ferrous ion?

Heat in a crucible a small crystal of potassium ferrocyanide until it turns black. When cold remove the solid, place it in a test tube, cover it with water and add a few drops of hydrochloric acid and warm the solution. Add a little of the solution to a solution of sodium carbonate. (4) What was formed? (5) What conclusion can you draw as to the presence of iron in the complex salt? (6) What did heating do to the complex salt? (7) How does a complex salt differ from a double salt?

Experiment 114. **Hydrolysis of Salts.**

Reference: **600.**

Discussion: Neutralization is the name given to the reaction between an acid and a base as the result of which a salt is formed. The reverse of this, the reaction of water with a salt to form an acid and a base, is called hydrolysis. A salt is hydrolyzed by water if the acid from which it is prepared is weak, that is, if it is but slightly ionized in water. The hydrolysis is brought about as the result of the union of the hydrogen ions furnished by the water and the negative ions of the acid to form the undissociated acid. For example, the salts of acetic acid, which is a relatively weak acid, are hydrolyzed.

The reactions which take place between the ions in a solution of sodium acetate may be indicated as follows:

$$\begin{array}{rcl} H_2O & \rightleftarrows & H^+ + OH^- \\ & & + \\ NaC_2H_3O_2 & \rightleftarrows & C_2H_3O^- + Na^+ \\ & & \downarrow\uparrow \\ & & HC_2H_3O \end{array}$$

The removal of hydrogen ions in this way leaves hydroxyl ions in the solution, which, accordingly, shows a basic reaction.

The salts prepared from weak bases are hydrolyzed in a similar way; in this case, however, as the result of the formation of the undissociated base, the solution shows an acidic reaction.

The extent to which hydrolysis of a salt takes place is determined (1) by the strength of the acid and of the base from which it is prepared, (2) the concentration of the solution, and (3) the temperature. The weaker the acid or base the greater the amount of hydrolysis. Hydrolysis also increases with increased dilution and with rise in temperature.

Whether an acid is weak or strong can be told by testing its aqueous solution with certain indicators. Litmus is very sensitive to hydrogen ions; such a weak acid as carbonic acid will change blue litmus paper to red. Congo red, on the other hand, requires a higher concentration of hydrogen ions to affect it; strong acids like hydrochloric acid change the dye to a deep blue color; it is not affected by carbonic acid.

Litmus is very sensitive to hydroxyl ions, and will show when hydrolysis takes place to only a very slight degree. When phenolphthalein changes from a colorless to a red compound a higher concentration of hydroxyl ions is required than in the case of litmus.

MATERIALS: Solutions of sodium chloride, sulphate, sulphide, carbonate, and acetate, of carbonic, acetic and concentrated hydrochloric acids, of calcium and ferric chlorides, of copper sulphate, and of phenolphthalein; litmus paper; Congo paper.

DIRECTIONS: (*a*) Determine by testing with litmus paper whether solutions of the following salts show a neutral, acidic, or basic reaction: sodium chloride, potassium nitrate, sodium sulphate, calcium chloride, sodium sulphide, copper sulphate, ferric chloride, sodium carbonate, and sodium acetate. **(1)** Tabulate the results and state in the case of each salt the strength (weak or strong) of the acid and the base from which the salt is derived.

(*b*) Test a dilute solution of hydrochloric acid with Congo red paper. **(1)** Tabulate the results of this and the following tests. Test a solution of carbon dioxide in water (carbonic acid) with blue litmus paper and with Congo red paper. **(2)**

Explain the result. Test a solution of hydrogen sulphide with the two indicators. (3) Explain the result. (4) Are these results in accord with the conclusions drawn from the experiments in (*a*) above?

Add 5 drops of glacial acetic acid to 10 c.c. of water, and 5 drops of concentrated hydrochloric acid to 10 c.c. of water. Test the two solutions with blue litmus paper and Congo red paper. (5) What are the relative strengths of hydrochloric acid and acetic acid?

(*c*) In a clean beaker boil for 15 minutes a filtered solution of 2 grams of copper sulphate dissolved in 50 c.c. of water. (1) Note carefully whether a small amount of solid separates. (2) Is the result in accord with the behavior of copper sulphate in (*a*) above? (3) What is the deposit? (4) What does the experiment show about the effect of rise in temperature on hydrolysis? (5) Give a reason for your answer.

(*d*) Test 10 c.c. of a solution of sodium acetate with pink litmus paper and with 3 drops of a solution of phenolphthalein. (1) Explain the results. Heat the solution just to boiling. (2) Explain the result. Cool the solution. (3) Explain the result.

(*e*) Add to 10 c.c. of a solution of ferric chloride a drop of a dilute solution of sodium hydroxide and shake the tube. Continue the addition of the alkali until after shaking a very slight precipitate remains. Filter the solution. Test it with litmus paper. (1) Is ferric chloride hydrolyzed? Add to the solution an equal volume of a solution of sodium acetate and heat to boiling. (2) What happened? Ferric acetate is first formed and then hydrolyzes. (3) Explain why in this case the hydrolysis is complete.

SODIUM AND POTASSIUM

EXPERIMENT 115. **The Action of Air Upon Sodium.**

Reference: Experiments 8 *a*, 20, 41.

DISCUSSION: A study of the action of air upon sodium brings together in review a number of facts which have already been taken up in previous experiments. These are the composition of the air, the action of certain metals with water, deliquescence, the action of carbon dioxide with bases, hydrates, efflorescence, and the formation of acid salts.

The great chemical activity of sodium is shown by the formation of the peroxide instead of the oxide when the metal is burned in the air.

MATERIALS: Sodium, starch iodide paper.

DIRECTIONS: PRECAUTION: *Handle sodium with the pincers. Do not allow water to touch it.* (*a*) Remove the oil from a small freshly cut piece of sodium by touching each side of the piece to filter paper. Scrape the coating from one side of the piece. **(1)** What is the appearance? **(2)** Is it permanent? **(3)** What is the hardness of sodium compared to common metals?

Place the piece of sodium on a watch glass and observe closely the changes taking place on its surface. **(4)** Recalling the composition of the air and a previous experiment in which you used sodium, what substance do you expect to see formed? **(5)** What evidence is there of the formation of this substance? **(6)** Write an equation for the reaction. **(7)** In what connection have you previously studied this reaction?

Put the sodium away on a shelf in your desk until the next exercise. **(8)** What is the appearance now? **(9)** What is the compound on the watch glass? **(10)** How do you account for its present state? **(11)** What is the technical name of the property which caused the substance to be in this condition?

Again set the watch glass and contents aside in your desk for one or more exercises, until the material appears as well

defined crystals. (12) Recalling once more the composition of the air, state what these crystals probably are. Transfer a small amount of the material to another watch glass and apply a simple test which would tend to corroborate your supposition, if true. (13) What did you do? (14) What was the result? (15) Write an equation for the reaction by which the crystalline substance was formed.

Put the remaining crystals back in your desk and examine them at each exercise until another distinct change has taken place. (16) What is now the appearance of the substance? (17) In what experiment have you observed a substance undergo a similar change in appearance? (18) What was the phenomenon called? (19) What further chemical reaction may this substance undergo if exposed to the air for a sufficiently long time?

(*b*) Place a piece of sodium on the inverted cover of a crucible and direct the flame of a burner down upon it until it ignites. When it has cooled, add a few drops of water and touch the solution with a piece of starch iodide paper, prepared as in Experiment 28. (1) What was the action on the paper? (2) What is thus proved to have been formed when the sodium burned? (3) Write an equation for the reaction. (4) What property of sodium does this reaction illustrate?

EXPERIMENT 116. **Baking Powder.**

Reference: **612.**

DISCUSSION: Baking powders are mixtures of sodium bicarbonate and some substance which, by producing an acid solution on the addition of water, will evolve carbon dioxide from the carbonate. Starch is also added to prevent reaction during storage and to dilute the baking powder, so that it may be evenly mixed with the flour when used. The choice of the acid-forming ingredient is dependent upon several considerations. It must be soluble enough to act quickly, but not too quickly; it must leave a final product of reaction which is not harmful; and the cost must be taken into account. The efficiency of the powder will depend also upon the thoroughness with which the ingredients are powdered and mixed.

MATERIALS: Sodium bicarbonate, potassium acid tartrate, sodium aluminium alum, primary calcium phosphate, starch, commercial baking powder.

DIRECTIONS: (*a*) Dissolve a quarter of a spatulaful of cream of tartar, $KH(C_4H_4O_6)$, in a test tube half full of warm water and test the solution with litmus paper. (**1**) What was the result? (**2**) To what class of compounds does it belong?

Repeat the experiment using primary calcium phosphate. (**3**) What was the action of the solution with litmus?

Likewise try the action of a solution of the alum of the composition $Na_2SO_4,Al_2(SO_4)_3,24H_2O$ with litmus. (**4**) What was the action? (**5**) What kind of process did the alum undergo in solution? (**6**) Write an equation for the reaction. (**7**) What properties make these substances suitable for making baking powders?

(*b*) (**1**) Write an equation for the reaction between cream of tartar and sodium bicarbonate. (**2**) Calculate the weight of of the former which will react with 5 grams of the latter, and (**3**) the weight of the carbon dioxide produced. (**4**) Calculate the weight of starch which must be added in order that the carbon dioxide shall be 12 per cent of the combined weights of the three ingredients.

Prepare the baking powder by thoroughly mixing in a mortar the ingredients in the amounts calculated above.

(**5**) Write an equation for the reaction taking place when alum baking powder acts. (**6**) Calculate the weight of alum necessary to use with 5 grams of sodium bicarbonate and the weight of starch to be added to make a mixture which will produce 12 per cent by weight of carbon dioxide. (**7**) Write an equation for the reaction in a phosphate baking powder and (**8**) calculate as above the weights of the ingredients to be used. (**9**) Calculate from the prices of the ingredients the cost of one pound of each kind of baking powder and record the prices of commercial baking powders.

(*c*) Add a pinch of your baking powder to a test tube half full of cold water and observe the rate of action. Repeat the experiment using hot water instead of cold. Try both of these experiments, using commercial baking powder instead of

your mixture. (1) What differences in rate of action did you observe? (2) How do you account for these differences?

EXPERIMENT 117. **Preparation of Sodium Carbonate.**

Reference: **610.**

DISCUSSION: As was recently stated in the experiment on the preparation of boric acid from borax, a compound may be obtained by double decomposition when it is sufficiently insoluble to be precipitated under the conditions of the reaction. Thus sodium acid carbonate, which is much less soluble than sodium chloride, will be precipitated from a saturated solution of sodium chloride upon the addition of ammonium acid carbonate. The ammonium acid carbonate, however, can not be added in solution, since the additional water would serve to keep the sodium acid carbonate also in solution. Therefore carbon dioxide is passed into a saturated solution of sodium chloride which contains ammonia.

MATERIALS: Sodium chloride, marble, lime water, phenolphthalein.

DIRECTIONS: (*a*) Saturate 10 c.c. of ammonium hydroxide solution with sodium chloride in a large test tube by adding an excess of the salt, shaking the mixture vigorously, and then filtering out the undissolved remainder. Set up a generator for carbon dioxide as in Experiment 38 *b*, and pass the gas into the solution until a heavy precipitate is obtained. Filter out the precipitate and dry it by pressing it between sheets of filter paper. (1) What is this substance? (2) Write two equations for the reactions which takes place and cause the formation of the precipitated substance. (3) What caused the second reaction to take place? (4) Why is not a solution of ammonium acid carbonate used to produce this reaction with a solution of sodium chloride?

(*b*) Treat a small portion of the precipitate in a test tube with 3 c.c. of water and add three drops of phenolphthalein. (1) What was the result?

Put into a crucible the remainder of the precipitate, or enough of it to one-quarter fill the crucible, and heat vigorously. While the substance is being heated, hold a drop of

lime water on a glass rod just above the crucible. (**2**) What appeared in the lime water? (**3**) What was being given off from the heated substance?

When bubbles no longer appear in the fused substance, allow the crucible to cool and add water, a cubic centimeter at a time, until the substance dissolves. (**4**) How much water was required? (**5**) How does the solubility compare with that of the unheated substance?

Add three drops of phenolphthalein to the solution. (**6**) What was the result? (**7**) Name three facts which show that reaction took place upon heating the substance in the crucible. (**8**) Write an equation for the reaction. (**9**) What industrial process is illustrated by this experiment as a whole?

EXPERIMENT 118. **Flame Tests.**

Reference: **615.**

DISCUSSION: When gases are heated to a sufficiently high temperature they emit colored light. The Bunsen flame is hot enough to volatilize the alkaline metals and alkaline earths and some of their compounds and thus to impart characteristic colors to the flame. All compounds of a given metal produce the same color, so that the appearance of the flame serves as a delicate test for that metal. Since the chlorides are the most volatile compounds, the material to be tested is usually treated with hydrochloric acid before it is put in the flame. The flame may be observed directly, or observed through a prism, which spreads out the light into separate colored bands.

Sodium compounds are so commonly present that the bright yellow color they produce in the flame often masks the delicate shades caused by potassium. Since sodium light, however, does not pass through blue cobalt glass, the flame may be viewed through a piece of this glass, and the color imparted by potassium may be detected, if it is present.

Colored fires may be produced by mixing with some easily combustible matter compounds which will give the desired color to the flame.

MATERIALS: Chlorides of sodium, potassium, calcium, strontium, and barium, calcium sulphate, powdered shellac, strontium nitrate, potassium chlorate, barium nitrate.

Directions: (*a*) Clean a platinum wire by alternately dipping it into concentrated hydrochloric acid in a test tube and holding it in the Bunsen flame, until no color is imparted to the flame by the wire.

Place a few crystals each of the chlorides of sodium, potassium, calcium, strontium, and barium on watch glasses or glass plates and moisten them with a few drops of concentrated hydrochloric acid.

Touch the clean wire to the sodium chloride and then hold it in the flame. (1) Record the color of the flame after this and each following trial. Clean the wire as before and repeat the experiment using potassium chloride instead of sodium chloride. Try the action again while viewing the flame through a piece of cobalt glass.

Wash your hands, draw the clean wire between your thumb and finger and then hold it in the flame. (2) What do you conclude as to the delicacy of the flame test for sodium compounds?

Touch the clean wire to both the sodium chloride and the potassium chloride and hold it in the flame. (3) Is the color caused by potassium observable? Repeat the experiment viewing the flame through cobalt glass. (4) How may potassium compounds be detected in the presence of sodium compounds?

Determine the colors imparted to the flame by the chlorides of calcium, strontium, and barium, respectively.

Moisten a little calcium sulphate with water and try the flame coloration produced by it. Repeat the trial with the calcium sulphate moistened with a little concentrated hydrochloric acid. (5) What difference in the flame did you observe? (6) What is the cause of this difference? (7) Why are chlorides to be preferred in making flame tests?

(*b*) Put into a small beaker half a spatulaful each of powdered shellac, strontium nitrate, and potassium chlorate. Pour the material back and forth from this into a second beaker several times until the substances are thoroughly mixed. Heap the mixture in an iron pan or on a brick in the hood and holding the burner at arms length let the flame play upon the mixture. (1) What happened? (2) What was the purpose

of the shellac? (3) Of the potassium chlorate? (4) What caused the color? (5) Why was strontium nitrate used instead of the chloride?

Repeat the experiment using barium nitrate instead of strontium nitrate. (6) What occurred?

EXPERIMENT 119. **Preparation of Potassium Nitrate.**

Reference: **619, 592.**

DISCUSSION: The source of the nitrates of commerce is Chili saltpeter, $NaNO_3$. Although potassium nitrate is a soluble salt it can be prepared from sodium nitrate by a double decomposition which takes place according to the reaction represented by the following equation:

$$NaNO_3 + KCl = KNO_3 + NaCl$$

The compound can be prepared in this way because potassium nitrate is but slightly soluble in water at low temperatures, whereas the other salts involved are much more soluble. In making potassium nitrate in this way equivalent quantities of sodium nitrate and potassium chloride are dissolved in such an amount of hot water that the sodium chloride formed in the reaction will remain in solution when the latter is cooled. The marked change in solubility of potassium nitrate with decrease in temperature and the relatively great solubility of sodium chloride in cold water assist materially in bringing about the metathesis.

MATERIALS: Sodium nitrate, potassium chloride.

DIRECTIONS: (*a*) To 22 grams of sodium nitrate and 19 grams of potassium chloride contained in a small beaker add 50 c.c. of water. Heat the mixture until the solution begins to boil. Filter, cover the solution with a piece of filter paper, and set it in your desk until the next exercise. (1) Describe the appearance of the crystals that separate. Filter off the crystals, and press them against the paper in the funnel to remove most of the mother liquor. When no more liquid drops from the funnel remove the beaker and set aside the filtrate and treat it as described in (*b*) below. Add 5 c.c. of cold

water to the crystals of potassium nitrate in the funnel and let them drain. Press the crystals between dry filter papers, and when they are dry, weigh them. (2) Record the weight.

(3) Calculate the weight of potassium nitrate equivalent to the sodium nitrate used. (4) What percentage of the theoretical amount was obtained in the experiment? (5) Why is the yield not the theoretical? (6) How could you calculate the largest percentage of potassium nitrate that could be obtained in the preparation as carried out? (7) If the solution were cooled to 0° would the yield of potassium nitrate be much greater? (8) Would the salt obtained be as pure? (9) Why?

(*b*) Heat the filtrate from the potassium nitrate crystals to boiling and evaporate it to about two-thirds its original volume. (1) What is the compound that separates during the boiling? Filter the hot solution rapidly. Save the filtrate. Press the crystals between filter papers. Examine them with a magnifying glass. (2) Do they resemble the crystals of potassium nitrate in appearance? (3) Upon what fact does the separation of potassium nitrate from the other three substances in solution depend? Examine the crystals that are deposited from the filtrate when it cools. (4) What are they?

CALCIUM, STRONTIUM, AND BARIUM

EXPERIMENT 120. **Hard Water.**

Reference: **628–631.**

DISCUSSION: Water which contains soluble compounds of calcium, magnesium or iron is called "hard water," because the addition of soap produces a precipitate by reaction with the compound in the water, instead of forming suds, as with pure water. The precipitate is usually a stearate formed by double decomposition between the compound in solution and sodium stearate, $NaC_{18}H_{35}O_2$, which is the chief constituent of ordinary soap.

The most common compound found in hard water is calcium acid carbonate, formed from limestone by the action of water containing carbonic acid. As was shown in Experiment 41 *b*, on the formation of calcium acid carbonate, this compound is decomposed by boiling the solution, when the calcium is precipitated as carbonate. The acid carbonates of magnesium and iron act similarly. Water containing any of these compounds is therefore called "temporary" hard water, in distinction from water containing other soluble salts of these metals, which is known as "permanent" hard water.

Temporary hard water may be made "soft" by removing the excess of carbonic acid, which preserves the acid carbonate from decomposition, by boiling the water, as stated above, or by neutralizing the acid with a base, such as ammonium hydroxide or calcium hydroxide. Both kinds of hard water may be "softened" by the addition of a reagent which will form an insoluble compound by reaction with the salt which is causing the hardness. Borax is often used for this purpose.

MATERIALS: Dilute soap solution, marble, lime water, solutions of calcium chloride, magnesium chloride, ferric chloride, calcium sulphate, sodium carbonate, and borax.

Directions: (*a*) Add 5 c.c. of soap solution to a test tube half full of distilled water and shake it vigorously. (1) Describe the appearance. (2) What is the soap commonly said to have produced?

(*b*) Prepare 60 c.c. of calcium acid carbonate solution as directed in Experiment 41 *b*. (1) Write equations for the reactions by which it was formed. (2) How is calcium acid carbonate formed in nature?

To 20 c.c. of the solution add 5 c.c. of soap solution and shake it. (4) How does the appearance of the liquid differ from that obtained in (*a*) after 10 seconds? (5) What name is given to any water which acts this way with soap? (6) Write an equation for the reaction which took place.

Add successive 5 c.c. portions of soap solution, shaking after each addition, until suds are formed which persist, after 10 seconds. (7) How many cubic centimeters of soap did you add in all? (8) How may the degree of hardness of water be determined?

Boil vigorously a fresh 20 c.c. portion of the solution of calcium acid carbonate for 5 minutes. (9) What was formed? (10) Write an equation for the reaction. Filter and shake the filtrate with 5 c.c. of soap solution. (11) What was the result? (12) Why did it differ from that obtained before the solution was boiled? (13) What is water called which behaves thus?

To the remainder of the original solution add ammonium hydroxide until an excess is present, as indicated by the odor. (14) What was precipitated? (15) Write an equation for the reaction. Filter, add 5 c.c. of soap solution and shake. (16) Was the water softened by the addition of the ammonium hydroxide? (17) What is the evidence? (18) What other compounds act as the calcium acid carbonate has in these experiments? (19) Write an equation to show why lime is used in the industries to soften hard water.

(*c*) To 20 c.c. each of solutions of calcium sulphate, calcium chloride, magnesium chloride, ferric chloride, and sodium chloride add 5 c.c. of soap solution and shake them. (1) State for each case whether or not suds were formed which remain permanent after 10 seconds. (2) Soluble salts of what elements

cause water to be hard? (**3**) How do these compounds make the water hard?

Boil 20 c.c. of a solution of calcium chloride. (**4**) Did a precipitate appear? Test the solution for hardness. (**5**) What did you do and observe? (**6**) Does boiling soften water which is hard on account of the presence of calcium chloride? (**7**) What is such hard water called?

To 20 c.c. of a solution of calcium sulphate add 10 c.c. of a solution of sodium carbonate. (**8**) Write an equation for the reaction taking place. Filter and test the filtrate for hardness. (**9**) What was the result?

Repeat the experiment using first borax solution instead of sodium carbonate and then again substituting ammonium hydroxide. (**10**) State for each case what happened upon the addtion of the reagent and (**11**) whether or not the water was softened by it. (**12**) How can permanent hard water be softened?

Experiment 121. **Plaster of Paris.**

Reference: **636.**

Discussion: Plaster of Paris is the hemihydrate of calcium sulphate, $(CaSO_4)_2,H_2O$, and is made by heating gypsum, $CaSO_4,2H_2O$, to about 145°. When the proper amount of water is added to plaster of Paris, the reverse action takes place and the dihydrate which is produced forms a rigid mass of interlocking crystals. The material expands slightly during this change and will thus completely fill all the small indentations in a mold in which it is put before it "sets." This expansion makes the material suitable for making casts.

Materials: Plaster of Paris, block of wood, vaseline, coin.

Directions: Mix five spatulafuls of plaster of Paris on a block of wood with enough water to make a soft dough. Press into the plaster a coin which has been covered with a thin coating of vaseline, and after 15 or 20 minutes, remove the coin and examine the cast. (**1**) How has the physical condition of the plaster changed? (**2**) What is this change called? (**3**) Are the indentations in the coin well marked in the cast? (**4**) To what property of the plaster is this due? (**5**) Write

an equation for the reaction taking place when plaster of Paris is prepared; (6) when it sets.

EXPERIMENT 122. **Comparison of Compounds of Calcium, Strontium and Barium.**

Reference: **589, 593, 642, 644, 651.**

DISCUSSION: The following study of compounds of the "alkaline earths" is made from the point of view of solubility and affords, in addition to a knowledge of the appearance of certain specific substances, a number of examples of the relation of ionization to precipitation. The underlying principle is that a reaction of double decomposition in solution tends to run to the formation of the least ionized substance. A small concentration of ions may be due to one of two causes, either to a small degree of dissociation, as in the case of ammonium hydroxide and of acetic acid, or to the insolubility of the compound, as in the case of barium sulphate.

If the equilibria involved are expressed in the following way, these relations become apparent:

$$\begin{array}{ccccc} AB & \rightleftarrows & A^+ & + & B^- \\ & & + & & + \\ CD & \rightleftarrows & D^- & + & C^+ \\ & & \uparrow\downarrow & & \uparrow\downarrow \\ & & AD & & CB \end{array}$$

When, for example, *AB* represents calcium chloride and *CD* sodium hydroxide, both highly ionized substancse, *AD* (calcium hydroxide) is precipitated, as it is the least ionized combination, due to its insolubility; but when *CD* represents ammonium hydroxide, no *AD* is precipitated, because the slight dissociation of *CD* in this case makes this compound the least ionized combination.

MATERIALS: Solutions of calcium, strontium, and barium chlorides, ammonium carbonate, ammonium oxalate, acetic acid, and sodium chromate, calcium sulphate, strontium sulphate.

DIRECTIONS: (1) Prepare a table for recording the results of these experiments, indicating the divisions into separate experiments by drawing horizontal lines across the table below

the reagents italicized in the following list, which constitutes the first column of the table: ***Reagents,*** sodium hydroxide, ***ammonium hydroxide, ammonium carbonate,*** ammonium oxalate, hydrochloric acid, ***acetic acid,*** sodium chromate, hydrochloric acid, ***acetic acid,*** sulphuric acid, hydrochloric acid, calcium sulphate, ***strontium sulphate.*** Head the second, third, and fourth columns calcium, strontium, and barium respectively and record in their proper places in the table your experimental results. When a precipitate appears, write "P" and name the color; when an insoluble substance reacts with a reagent write "R"; and when nothing is observable make a dash.

(*a*) Add 5 c.c. of sodium hydroxide solution to each of three test tubes containing 5 c.c. of solutions of calcium chloride, strontium chloride, and barium chloride respectively. Repeat the experiments substituting ammonium hydroxide for the sodium hydroxide. (**2**) Make a diagram, of the kind given in the discussion, to show the equilibria involved in the addition of sodium hydroxide to calcium chloride and one involved in the addition of ammonium hydroxide to calcium chloride. Underline in each, the least ionized substance and state the causes of the small concentration of ions. (**3**) Why does sodium hydroxide produce a different result from ammonium hydroxide?

(*b*) To 5 c.c. each of solutions of calcium, strontium, and barium chlorides add a few drops of ammonium hydroxide, to insure the alkalinity of the solution, and then 5 c.c. of ammonimun carbonate solution. (**2**) Construct a diagram for the equilibria in the case of calcium chloride and ammonium carbonate. (**3**) Why does the addition of ammonium carbonate give a different result from the addition of ammonium hydroxide?

(*c*) To 5 c.c. each of solutions of calcium, strontium, and barium chlorides, made alkaline with a few drops of ammonium hydroxide, add 5 c.c. of ammonium oxalate. Pour half the contents of each test tube, including a share of the precipitates, into three other test tubes. Add an excess of hydrochloric acid to each of these test tubes and an excess of acetic acid to each of the original tubes. (**2**) Make equilibria diagrams

for the cases of calcium oxalate and hydrochloric acid, calcium oxalate and acetic acid and barium oxalate and acetic acid. (3) Why does hydrochloric acid differ from acetic acid in its action with calcium oxalate? (4) Why does acetic acid differ in the action with calcium oxalate and barium oxalate? (5) How may a soluble calcium salt be distinguished from a barium or strontium salt?

(*d*) To slightly alkaline solutions of the three chlorides, as in (*c*), add 5 c.c. portions of sodium chromate solution. Divide the contents of the test tubes and try the action of hydrochloric acid and of acetic acid on each precipitate, as before in (*c*). (2) Make equilibria diagrams for the cases of calcium chromate and acetic acid and of barium chromate and acetic acid. (3) Why does acetic acid act differently with these two chromates? (4) How may a soluble barium salt be distinguished from a soluble calcium or strontium salt?

(*e*) To 5 c.c. each of solutions of calcium, strontium, and barium chlorides add 1 c.c. of dilute sulphuric acid. Determine whether or not the sulphates formed will react with hydrochloric acid.

To a test tube half full of water add about 1 gram of solid calcium sulphate, shake the mixture vigorously and then filter it. To half of the filtrate add 5 c.c. of strontium chloride solution and to the other half 5 c.c. of barium chloride solution. (2) Explain why the reaction took place in each case.

Shake a mixture of 5 c.c. of water and 1 gram of strontium sulphate and filter. To the filtrate add 5 c.c. of barium chloride solution. (3) What are the relative solubilities of the three sulphates studied? (4) How do these experiments establish this order? (5) Where in the periodic classification do the elements studied in this experiment occur? (6) What is this group called?

(*f*) Determine as in Experiment 118 the colors imparted to the flame by the chlorides of these elements. (1) Record the results.

MAGNESIUM, ZINC, AND MERCURY

EXPERIMENT 123. **Magnesium Nitride.**

Reference: **318.**

DISCUSSION: When magnesium burns in the air, the chief product is magnesium oxide, which is formed with the evolution of much energy, as light and heat. Under these conditions the usually inert nitrogen of the air becomes sufficiently active to unite with some of the magnesium. The resulting magnesium nitride is decomposed by water.

MATERIALS: Magnesium powder, copper wire, litmus paper.

DIRECTIONS: Put half a spatulaful of powdered magnesium into a crucible supported on a clay triangle. Heat the crucible vigorously and then ignite the magnesium by directing the flame of the burner into the crucible. Continue to heat the crucible and stir the burning magnesium with a piece of copper wire until no further ignition takes place. When the crucible has cooled, allow a few drops of water to fall upon the contents and at the same time lay a piece of pink litmus paper across the top of the crucible. (1) What was the action on the litmus paper? (2) To what substance is the action due? (3) Write equations for the reactions which took place when the magnesium burned. (4) Which was the chief reaction? (5) How did this facilitate the other reaction? (6) Write equations for the two reactions which took place upon the addition of water.

EXPERIMENT 124. **Magnesium Chloride.**

Reference: **656.**

DISCUSSION: The great solubility of magnesium chloride, which accounts for its deliquescence, and the extensive hydrolysis which takes place upon heating a solution of this salt, are shown in the following experiment.

MATERIALS: Magnesium chloride, litmus paper.

DIRECTIONS: Put half a spatulaful of magnesium chloride

into a 6-inch test tube and add 2 c.c. of water. (1) What is shown as to the solubility of the substance?

Boil away all of the water in the test tube and test the vapor with litmus paper as it issues from the tube. (2) What was the result? (3) What property of magnesium chloride is thus shown? Heat the residue in the tube strongly for two or three minutes and when it is cold, add about 3 c.c. of water to the residue. (4) Did it dissolve completely? (5) How do you account for this?

EXPERIMENT 125. **Zinc Hydroxide.**

Reference: **595, 663.**

DISCUSSION: Zinc hydroxide forms both hydroxide ions and hydrogen ions in solution, that is, it may act either as a base or as an acid. These facts are shown in the following expression:

$$\begin{array}{ccccc} 2H^+ + ZnO_2^{--} & \rightleftarrows & Zn(OH)_2 & \rightleftarrows & Zn^{++} + 2OH^- \\ & & \downuparrows & & \\ & & Zn(OH)_2(\text{solid}) & & \end{array}$$

If an acid, as hydrochloric acid, is added to zinc hydroxide, the hydrogen ions of the acid unite with the hydroxyl ions from the zinc hydroxide and cause the reaction to proceed to the right in the expression just given. This action causes solid zinc hydroxide to dissolve to replace that originally in solution, and a solution of zinc chloride is the final product. If, on the other hand, a base, as sodium hydroxide, is added to the zinc hydroxide, the hydroxyl ions of the base unite with the hydrogen ions from the zinc hydroxide, the reaction proceeds to the left, the solid zinc hydroxide dissolves as before, but the final product is sodium zincate.

The solubility of zinc hydroxide in ammonium hydroxide is due to a third cause. Ammonia forms by direct union with the zinc ion a complex ion of the composition $Zn(NH_3)_4{}^{++}$. The formation of these ions removes zinc ions from solution. This causes the reaction again to proceed to the right as indicated in the expression above, with the consequent solution of the solid zinc hydroxide.

In summary, any reagent which will remove one of the ions formed by zinc hydroxide will cause the solid to disappear. If the reagent is an acid, the hydroxyl ions are removed, if it is a base, the hydrogen ions, and if it is ammonia, the zinc ions.

MATERIALS: Solution of zinc sulphate.

DIRECTIONS: (*a*) To 10 c.c. of zinc sulphate solution in a test tube add sodium hydroxide solution drop by drop until the liquid contains a considerable precipitate. (1) What is the precipitate? (2) Write an equation for its formation.

Shake the liquid and pour half of it into another test tube. To one portion add hydrochloric acid and to the other sodium hydroxide, until a distinct change takes place. (3) What occurred in each case? (4) Write equations for the reactions. (5) As what kind of a compound did the zinc hydroxide act when treated with the acid? (6) When treated with the base? (7) Write an expression to show how zinc hydroxide ionizes. (8) Explain with reference to this expression why the reaction with the acid and with the base took place.

(*b*) To 10 c.c. of zinc sulphate solution add a few drops of ammonium hydroxide. (1) What is formed? (2) Write an equation for the reaction.

Add to the test tube ammonium hydroxide until a distinct change takes place. (3) What occurred? (4) What compound is present in a solution of ammonium hydroxide in addition to the ammonium hydroxide? (5) Write an equation for the reaction which occurred. (6) Explain with reference to the expression for the ionization of zinc hydroxide why the reaction took place. (7) Is the solution of the zinc hydroxide in this case due to the same cause as the solution of magnesium hydroxide on the addition of ammonium salts, as observed in Experiment 106 *b*? (8) Wherein lies the resemblance or difference? (9) Upon what general principle does the reaction of zinc hydroxide with acids, bases, and ammonia depend in all three cases?

EXPERIMENT 126. **Zinc Chloride.**

Reference: **664.**

DISCUSSION: It was shown in Experiment 125 that the basic

properties of zinc hydroxides are very weak and it therefore follows that soluble zinc salts will become hydrolyzed in solution. If the conditions are such that the acid formed by this hydrolysis can pass off, the hydrolysis will proceed, leaving a basic salt.

MATERIALS: Zinc chloride.

DIRECTIONS: To 5 c.c. of water in a small test tube add several crystals of zinc chloride. Test the solution with litmus paper. (1) What was the result? (2) What does this prove concerning the action of zinc chloride with water? (3) Did any precipitate appear? (4) What does this fact show as to the extent of the action?

Boil the solution, and when most of the water has evaporated, test the steam at the mouth of the tube with blue litmus paper. (5) What was the result? (6) What caused the action? (7) What was its source?

Continue the evaporation to dryness and heat the residue in the test tube to as high a temperature as the glass will permit for several minutes. When the tube has cooled add 5 c.c. of water. (8) Is the substance in the tube soluble? Add 5 c.c. of nitric acid; heat to boiling; filter, if necessary; and test the solution for a chloride? (9) What was the result of the test? (10) What was formed by heating magnesium chloride?

EXPERIMENT 127. **Zinc Sulphide. A Test for Zinc.**

Reference: **666.**

DISCUSSION: Zinc sulphide is characterized by being the only common white sulphide and by being insoluble in acetic acid but soluble in more active acids, such as hydrochloric or sulphuric acids. These characteristics may be made use of in recognizing compounds of zinc.

MATERIALS: Solutions of zinc sulphate, of ammonium sulphide, of acetic acid, and hydrogen sulphide.

DIRECTIONS: (*a*) To 10 c.c. of zinc sulphate solution in a test tube add 10 c.c. of ammonium sulphide. (1) What is the appearance and the solubility of zinc sulphide? (2) Write an equation for the reaction of its formation.

Filter out the precipitate, wash it on the paper with 5 c.c. of water, transfer the funnel to a clean test tube, poke a hole in the tip of the filter paper, and wash the precipitate into the test tube with 15 c.c. of water. Pour a third of the suspended zinc sulphide into each of two other test tubes. To one add 10 c.c. of hydrochloric acid, to the other 10 c.c. of sulphuric acid and to the remainder of the zinc sulphide 10 c.c. of acetic acid. (3) What happened in each case? (4) Write expressions similar to those used in Experiment 122 to show the equilibria involved between zinc sulphide and acetic acid. (5) Explain with reference to these expressions the difference in action of the acids with zinc sulphide.

(*b*) Bubble hydrogen sulphide into 10 c.c. of zinc sulphate solution in a test tube until the solution smells strongly of hydrogen sulphide. (1) What insoluble substance was formed? (2) Write an equation for its formation.

Filter out the precipitate and add a few drops of ammonium hydroxide to the filtrate. (3) What appeared? (4) Was all the zinc precipitated by the hydrogen sulphide? (5) What is the evidence? (6) Why was the reaction incomplete? (7) Why did it take place at all? (8) What reagent should be used to precipitate all the zinc as sulphide?

(*c*) Prepare 10 c.c. of sodium zincate solution as in Experiment 125. (1) What did you do? To this add 5 c.c. of ammonium sulphide. (2) What was formed? (3) Write an equation for the reaction.

(*d*) (1) State what you would do in testing for zinc, and what would happen if it were present.

EXPERIMENT 128. **Compounds of Mercury.**

Reference: **672, 674, 676, 677, 679, 681.**

DISCUSSION: The important reactions of the compounds of mercury depend upon the following facts: (1) Two sets of compounds exist, in which mercury appears with the valences of one and of two respectively. Some reagents may reduce mercuric compounds with the valence of two, to mercurous compounds with the valence of one, or may even reduce them to metallic mercury, as in the case of the chlorides. Some

mercurous compounds, on the other hand, undergo spontaneous oxidation to mercuric compounds, with the necessary separation of metallic mercury, as in the case of the sulphide.

(2) Hydroxides of mercury are unstable and consequently an oxide is precipitated where a hydroxide would be expected as a product of double decomposition. Moreover, the oxides of mercury do not react with an excess of a base to form salts, as does zinc hydroxide.

(3) Mercuric salts react with ammonia to form ammono basic compounds. This is a type of reaction not heretofore studied. It will be recalled that in the hydrolysis of a salt the hydroxide radical of water unites with the metal of the salt and the remaining hydrogen unites with the negative radical of the salt; but that in the case of many salts, this action is incomplete, because the substitution of only part of the negative radicals by hydroxide takes place. Thus, mercuric chloride can be hydrolyzed in accordance with the expression: $HgCl_2 + H_2O = Hg(OH)Cl + HCl$. Just as water thus reacts as a compound of hydrogen with the hydroxide radical, so ammonia may react as a compound of hydrogen with a radical of the composition NH_2; and in the same way that many salts react with water in hydrolysis, so mercuric salts react with ammonia in ammonolysis. The resulting compound is analogous in composition to the basic salt, with the substitution of the radical NH_2 for the radical OH. These compounds must not be confused with the complex salts formed by the direct union of ammonia to salts of zinc, copper, and other metals.

MATERIALS: Solutions of mercuric chloride, of mercurous nitrate, and of stannous chloride, and hydrogen sulphide.

DIRECTIONS: (*a*) Dilute 5 c.c. of mercuric chloride solution with an equal volume of water in a test tube and add to it ten drops of stannous chloride solution. (1) State the chemical name, the commercial name, the color, and the solubility of the mercury compound formed. (2) Write an equation for the reaction. (3) What kind of a process has the mercuric chloride undergone? (4) What was the agent?

To the contents of the test tube add more stannous chloride until another distinct change takes place. (5) Describe the

change. (6) Write an equation for the reaction. (7) What kind of a change has the mercury compound undergone in this case?

(*b*) Bubble hydrogen sulphide into 5 c.c. of mercuric chloride solution. (1) Name and describe the appearance of the mercury compound formed. (2) Write an equation for its formation. Add to it 5 c.c. of concentrated nitric acid and heat it to boiling. (3) What do you conclude as to the solubility of mercuric sulphide? (4) Upon what do you base your conclusion?

Bubble hydrogen sulphide into 5 c.c. of mercurous nitrate solution. (5) What appeared? This is a mixture of mercuric sulphide and mercury. (6) What would have been formed by double decomposition? (7) What does this experiment show concerning the stability of mercurous sulphide? (8) What process has the mercury undergone in changing from mercurous nitrate to mercuric sulphide? (9) What was reduced?

(*c*) To 5 c.c. of mercuric chloride solution add ten drops of sodium hydroxide solution. (1) Name and describe the precipitate. (2) Write an equation for the reaction. Add sodium hydroxide in excess. (3) What happened?

Repeat the experiment using mercurous nitrate instead of mercuric chloride. (4) Name and describe the precipitate. (5) Write an equation for its formation. (6) Did it react with an excess of sodium hydroxide? (7) Does mercury show properties of an acid-forming element? (8) Upon what do you base your answer? (9) What other element in this group of the periodic classification differs from mercury in this respect?

(*d*) To 5 c.c. of mercuric chloride solution add 5 c.c. of ammonium hydroxide solution. (1) What is the name, color, and solubility of the mercury compound formed? (2) With what substance in the ammonium hydroxide solution did the mercuric chloride react? (3) To what type of reaction previously studied is this type of reaction similar? (4) Explain with the use of two equations where the similarity exists.

Prepare some mercurous chloride by adding 5 c.c. of dilute hydrochloric acid to 5 c.c. of mercurous nitrate, filter, and add

a few drops of ammonium hydroxide solution to the precipitate on the paper. (5) What happened? The substance on the paper is a mixture containing ammono-basic mercuric chloride and mercury. (6) To what is the color due? (7) In what other reaction studied in this experiment did a similar spontaneous oxidation and reduction take place? (8) Write an equation for the reaction.

(*e*) (1) What characteristic action of a mercuric salt would you choose as suitable for the basis of a test? (2) Describe the procedure in making the test and the results if a mercuric salt were present. (3) What reaction would be suitable as a test for a mercurous salt? (4) Describe the procedure and the result of making a test if a mercurous salt were present.

ALUMINIUM

EXPERIMENT 129. Aluminium Hydroxide.

Reference: **685.**

DISCUSSION: In some of the preceding experiments, the importance has been emphasized of studying the behavior of the hydroxides of the elements with acids, with sodium hydroxide, and with ammonium hydroxide, as showing the base- or acid-forming properties of the elements, their ability to form complex or substituted compounds with ammonia, and the means of distinguishing compounds of one element from those of others. The following experiments on aluminium hydroxide are designed to show the chemical properties of this element in these same respects.

MATERIALS: Solution of aluminium sulphate.

DIRECTIONS: To 10 c.c. of aluminium sulphate solution in a test tube add 10 drops of sodium hydroxide solution. (1) Describe the appearance of the precipitate. (2) Write the equation for its formation.

Pour one-third of the contents of the test tube into each of two other test tubes, to one of which add an excess of hydrochloric acid. (3) What occurred? (4) Write an equation for the reaction. (5) As what kind of a compound does aluminium hydroxide act?

To the second of the three test tubes add an excess of sodium hydroxide. (6) What occurred? (The salt formed is that derived from a dehydrated aluminium hydroxide of the composition $Al(OH)_3 - H_2O = HAlO_2$.) (7) Write an equation for the reaction. (8) As what kind of a compound does aluminium hydroxide act in this case? (9) Write an expression of the kind used in Experiment 125 to show the equilibria involved in a suspension of aluminium hydroxide. (10) Explain with reference to this expression the actions of hydro-

chloric acid and of sodium hydroxide with aluminium hydroxide.

To the third test tube add an excess of ammonium hydroxide. **(11)** What occurred? **(12)** Does aluminium hydroxide form either complex compounds or substituted compounds with ammonia?

(13) By the use of what reagent or reagents can a soluble aluminium salt be distinguished from a soluble calcium salt? State what happens with the reagent in each case? **(14)** Likewise how can aluminium salts be distinguished from magnesium salts? **(15)** From zinc salts? **(16)** From mercury salts?

EXPERIMENT 130. Method for Cleaning Silver.

Reference: **567.**

DISCUSSION: It was shown in Experiment 97 that when a metal is put into a solution with which it will react with the evolution of hydrogen, and when also this metal is in contact with another metal below it in the electromotive series, the hydrogen appears on the surface of the less active metal and the more active metal goes into solution as a compound. Thus, when aluminium in contact with silver is treated with sodium hydroxide, hydrogen appears upon the silver and sodium aluminate is formed. If the silver is coated with the sulphide, the usual tarnish on silver ware, the hydrogen will reduce this sulphide. Use is made of these reactions in cleaning tarnished silver.

MATERIALS: Solution of sodium carbonate; strips of aluminium, and of tarnished silver to be returned after using.

DIRECTIONS: Heat a strip of aluminium, about one by three centimeters in size, with enough sodium carbonate solution to cover it in a test tube. **(1)** What is the evidence of chemical action? **(2)** How do you account for this action of sodium carbonate with aluminium?

Slip into the test tube a strip of tarnished silver so that it will *touch* the piece of aluminium and allow only enough solution to remain in the tube so that but half the strip of silver is submerged. Heat the solution to boiling, remove the test tube from the flame and observe the piece of silver. **(3)** What

is the gas set free on its surface? (4) What relation has this phenomenon with the relative positions of aluminium and silver in the electromotive series? Take the silver from the tube and compare the appearance of the two ends of the strip. (5) How has the part which was in the solution changed? (6) Of what does silver tarnish consist? (7) Write an equation for the reaction which took place when it disappeared. (8) How can this action be made of practical household use?

TIN AND LEAD

EXPERIMENT 131. Hydroxides of Tin.

Reference: **698–700.**

DISCUSSION: As in the case of other elements recently studied, a knowledge of the reaction of its hydroxides is important in summarizing the properties of tin in respect to its electropositive or electronegative characteristics. The position of tin in the fourth group of the periodic classification would lead to the prediction of distinctly acidic properties for stannic hydroxide, yet the fact that tin occurs so far down as the sixth member of the group would indicate the possibility of some basic characteristics.

Stannic hydroxide can be prepared from the chloride by precipitation by a base or it may be formed by oxidation of the metal in presence of water, when a hydrated oxide results. The former compound is called α-stannic acid, the latter β-, or meta stannic acid.

MATERIALS: Solution of stannic chloride, concentrated nitric acid.

DIRECTIONS: (*a*) Dilute 5 c.c. of stannic chloride solution with an equal volume of water in a test tube and add sodium hydroxide solution, a few drops at a time, until a precipitate is formed. (1) Describe the appearance of this precipitate. (2) What is it called? (3) Write an equation for its formation.

Pour half of the suspended precipitate into another test tube and add an excess of hydrochloric acid to one tube and an excess of sodium hydroxide to the other. (4) What happened in each case? (5) Write equations for the reactions. (6) How might these reactions have been predicted from the position of tin in the periodic classification?

(*b*) Add 5 c.c. of concentrated nitric acid to a small piece of tin in a test tube in the hood. Warm the tube, if necessary, to start the reaction and then stand it in a gas bottle to

catch any liquid which may overflow. When the action has ceased, dilute the solution with three times its volume of water and filter. Wash the precipitate with three 5 c.c. portions of water and then transfer it to a clean test tube by puncturing the filter paper. (1) What gas was evolved in the reaction? (2) What do you conclude as to the rôle of nitric acid? (3) What was oxidized? (4) What is the composition of the precipitate? (5) What is it called?

Pour half of the suspended precipitate into a clean test tube and treat one portion with hydrochloric acid and the other with sodium hydroxide. (6) What happened in each case? (7) The oxide of what other element in the same group of the periodic classification as tin behaves in the same way with solutions of acids and bases?

(*c*) To 5 c.c. of stannous chloride solution add a solution of sodium hydroxide very slowly, and finally in excess. (1) What were the results obtained? (2) Write equations for the reactions.

Boil the solution until a precipitate appears. Metallic tin and sodium stannate are formed. (3) Write an equation for the reaction.

EXPERIMENT 132. **Chlorides of Tin.**

Reference: **701.**

DISCUSSION: As would be predicted from the position of tin in the electromotive series, non-oxidizing acids will react with this metal to form salts and evolve hydrogen. If hydrochloric acid is used, mercuric chloride can be used to determine whether stannous chloride, or stannic chloride is produced. This is the same reaction which was employed in the reduction of mercuric chloride to mercurous chloride, studied in Experiment 128 *a*.

Suitable reagents can oxidize or reduce stannous chloride or stannic chloride, respectively, so that conversely these compounds can be used for reduction and oxidation. Since the oxygen of the air will slowly oxidize stannous chloride, special means must be used to preserve solutions of this compound in the laboratory.

MATERIALS: Tin, solutions of mercuric chloride, of ferric chloride, concentrated hydrochloric acid, concentrated nitric acid.

DIRECTIONS: Pour upon a small piece of tin in a test tube 5 c.c. of concentrated hydrochloric acid and 5 c.c. of water. Heat the solution if necessary and keep it at such a temperature that vigorous action continues until the tin has disappeared. (1) What was the gas evolved? (2) Why might you expect that it would be formed?

To 5 c.c. of mercuric chloride solution add a few drops of this solution. (3) What is the substance which appears? (4) What does this prove as to the tin compound formed by the original action of the hydrochloric acid? (5) Write an equation for the reaction.

(Ferric salts are reddish brown and ferrous salts colorless in dilute solutions.) Dilute 1 c.c. of ferric chloride solution with 3 c.c. of water and add some of your original solution drop by drop till a change of color occurs. (6) What was the change in color? (7) Write an equation for the reaction. (8) What kind of change has the tin compound undergone? (9) As what kind of an agent has it acted?

To 5 c.c. of the original solution add 10 drops of concentrated nitric acid and heat to boiling. Cool the solution and test for a stannous salt. (10) How did you test? (11) With what result? (12) What has happened to the stannous chloride? (13) Will this reaction be brought about by the air? (14) What is used in the laboratory to preserve solutions of stannous chloride from change?

EXPERIMENT 133. **Sulphides of Tin.**

Reference: **478, 702.**

DISCUSSION: The sulphides of tin are chiefly of interest in relation to their use in separating tin from other metals and in identifying tin in the course of qualitative analysis. Their important properties are the insolubility in dilute acid, and the solubility in ammonium polysulphide and concentrated hydrochloric acid.

MATERIALS: Solutions of stannic chloride, of stannous

chloride, of ammonium polysulphide, concentrated hydrochloric acid, and hydrogen sulphide.

DIRECTIONS: (*a*) Bubble hydrogen sulphide into 10 c.c. of a solution of stannic chloride. (**1**) Describe the precipitate. (**2**) Write an equation for its formation.

Filter out the precipitate and wash it with three portions of water of 5 c.c. each. After puncturing the tip of the filter paper, wash the precipitate into a clean test tube with as little water as possible. Transfer half of the precipitate to another test tube and treat it with 5 c.c. of ammonium polysulphide. (**3**) What happened? (**4**) Write an equation for the reaction. (**5**) The sulphides of what other elements react in a similar way?

To the remainder of the precipitate add 5 c.c. of concentrated hydrochloric acid and heat to boiling. (**6**) What happened? (**7**) Referring to the properties of the sulphides of arsenic studied in Experiment 89, state how stannic sulphide may be distinguished from them.

(*b*) Bubble hydrogen sulphide through 5 c.c. of stannous chloride solution. (**1**) What is the appearance of the precipitate? (**2**) Write an equation for its formation.

Filter, wash, and treat the precipitate with ammonium polysulphide as in part (*a*) above. (**3**) State what happened. (**4**) Write an equation for the reaction.

Treat the solution with dilute hydrochloric acid. (**5**) What is the precipitate? (**6**) How do you recognize it? (**7**) What kind of change has the tin undergone during these reactions? (**8**) What was the agent?

EXPERIMENT 134. **Oxides of Lead.**

Reference: **706.**

DISCUSSION: Since lead exhibits two valences in its compounds, two different oxides, lead monoxide and lead dioxide. would be anticipated. In addition to these, however, a third oxide, red lead, is known, the constitution of which is to be determined in a part of this experiment.

MATERIALS: Lead, oxides of lead, concentrated hydrochloric acid, starch iodide paper, bromine water, charcoal.

Directions: (*a*) Place about one gram of lead in a shallow hole in a piece of charcoal and heat it in the *oxidizing* flame of a blow pipe until a deposit is formed about the hole. (1) What is the color of this deposit? Compare it with the samples of oxides of lead in the laboratory. (2) What is it?

(*b*) To 5 c.c. of lead nitrate solution add sodium hydroxide solution until a considerable precipitate is formed. To this add bromine water drop by drop until the precipitate undergoes a distinct change. (1) What is the appearance of the new compound? Compare it with the samples of the oxides of lead. (2) What is it? (3) Write an equation for its formation. (4) What kind of a change has the lead undergone? (5) What was the agent?

(*c*) Warm half a spatulaful of lead dioxide with 5 c.c. of concentrated hydrochloric acid in a test tube and smell cautiously at the mouth of the tube. (1) What do you conclude was formed in the test tube?

Add 10 c.c. of water and filter. When about 5 c.c. of filtrate have been collected, add a few drops of dilute sulphuric acid. (2) What appeared? (3) What is the substance? (4) Write an equation for the reaction taking place in making the test. (5) What kind of a change has the lead of the lead dioxide undergone? (6) As what kind of agent has it acted? (7) Upon what did it thus act? (8) Write an equation for this reaction.

(*d*) Warm a quarter of a spatulaful of red lead with 10 c.c. of dilute nitric acid in a test tube, and when no further action takes place, filter the liquid. (1) What is the appearance of the residue? (2) What do you take it to be? (3) Judging by the inactivity of this substance with dilute acid, does lead here exhibit acid-forming or base-forming properties?

To the filtrate add a few drops of sulphuric acid. (4) What did the filtrate contain? (5) State in logical order the steps by which you came to this conclusion. (6) To what class of elements does lead appear to belong in this compound?

(7) What is the name and formula of the acid of which lead dioxide is the anhydride? (8) Of the lead salt of this acid? (9) What is the evidence that red lead is this compound?

(10) Write an equation for the reaction between red lead and nitric acid.

EXPERIMENT 135. **Preparation of Lead Dioxide.**

Reference: **706.**

DISCUSSION: When a lead salt in which the metal has the valence 2 is treated with an alkaline solution of a hypochlorite or a hypobromite, oxidation takes place and lead dioxide is precipitated. In the preparation described below the hypobromite is prepared by dissolving bromine in a cold solution of sodium hydroxide:

$$2NaOH + Br_2 = NaOBr + NaBr + H_2O$$

To this solution which contains an excess of sodium hydroxide is added a solution of lead acetate. The equation for the reaction which takes place is as follows:

$$Pb(C_2H_3O_2)_2 + NaOBr + 2NaOH = PbO_2 + 2NaC_2H_3O_2 + NaBr + H_2O$$

MATERIALS: Bromine, crystalline lead acetate, sodium hydroxide.

DIRECTIONS: Dissolve 17 grams of sodium hydroxide in 200 c.c. of water, and add to the cooled solution 5 c.c. of bromine. Dissolve 34 grams of lead acetate, $Pb(C_2H_3O_2)_2,3H_2O$, in 200 c.c. of hot water and cool the solution. (If a small amount of substance remains insoluble it will not affect the result.) Add the solution of sodium hypobromite to the solution of lead acetate in a large beaker, and heat the mixture to boiling for 5 minutes. Let the solid settle and then pour off carefully most of the liquid. Fill the beaker with hot water nearly to the top, heat to boiling, let the solid settle and decant the liquid as before. Repeat the washing twice in this way. Filter off the lead dioxide, wash it twice with about 100 c.c. of boiling water, and set it aside to dry.

Report (1) the weight of the dry dioxide, (2) the theoretical weight calculated from the weight of lead acetate used, and (3) the percentage of this obtained in the preparation.

EXPERIMENT 136. **Salts of Lead.**

Reference: **707, 709, 710.**

DISCUSSION: Many lead salts are insoluble and thus can be prepared by precipitation from solution. A knowledge of their appearance is important in identifying compounds of lead.

MATERIALS: Solutions of lead nitrate, and of potassium or sodium iodide, hydrogen sulphide.

DIRECTIONS: (*a*) Dilute 3 c.c. of a solution of lead nitrate with 5 c.c. of water and bubble hydrogen sulphide into the solution. (1) Name and describe the precipitate. (2) Write an equation for its formation. Add 5 c.c. of dilute nitric acid. (3) Does the precipitate dissolve in nitric acid?

(*b*) To 3 c.c. of lead nitrate diluted with 5 c.c. of water add 10 drops of dilute hydrochloric acid. (1) Name and describe the precipitate. (2) Write an equation for its formation. Heat the solution. (3) What change occurs? Cool it again. (4) What appears? (5) What do you conclude about the solubility of lead chloride? (6) What other insoluble chlorides have you made? (7) How does lead chloride differ from them?

(*c*) To 3 c.c. of lead nitrate diluted as before add 3 c.c. of potassium or sodium iodide solution. (1) Write an equation for the reaction. Heat the solution to boiling. (2) What happened? Cool the solution again. (3) Name and describe the precipitate.

(*d*) To 3 c.c. of lead nitrate diluted as before add 10 drops of dilute sulphuric acid. (1) Name and describe the precipitate. (2) Compounds of what other metals show a similar result upon the addition of sulphuric acid? (3) From the foregoing experiments select two reactions which in conjunction can be used to identify compounds of lead in solution. (4) State the procedure in testing for lead.

SILVER AND COPPER

EXPERIMENT 137. **Preparation of Metallic Silver.**

Reference: **714.**

DISCUSSION: Silver may be prepared from its compounds by displacement by a metal above it in the electrochemical series, or by a process of reduction. Numerous reducing agents may be used in the latter case in solution, or the reaction may be accomplished by heating the silver compound in a reducing flame.

MATERIALS: Ten cent pieces cut in quarters, concentrated nitric acid, mercury, anhydrous sodium carbonate, charcoal.

DIRECTIONS: (*a*) Place a quarter of a 10 cent piece in a 100 c.c. beaker, pour upon it 5 c.c. of water and 5 c.c. of concentrated nitric acid. When the metal has disappeared, pour a few drops of the solution into a test tube and add ammonium hydroxide very slowly until an excess is present. (1) What is the color of the solution? (2) For what is this a test? (3) With what metal is coinage silver alloyed?

(*b*) Put 1 c.c. of mercury in an evaporating dish and add about a third of the solution in the beaker. Set the dish aside in your desk until the next exercise, when the following questions should be answered. (1) Describe the substance deposited on the mercury. (2) What is it? (3) Write an equation for its formation. (4) State why this reaction takes place.

Remove the crystals with pincers and pour the mercury into the bottle prepared to receive it. Take great care that no mercury gets into the sink. (5) Why?

(*c*) Heat the solution remaining in the beaker and add dilute hydrochloric acid to it slowly until no more precipitation takes place. Add hot water until the beaker is nearly full, stir the precipitate, and when it has settled, pour off as much of the water as possible without losing any of the precipitate.

Wash the precipitate three times with hot water in this way and finally transfer it to a filter. Spread out the filter paper, scrape off the silver chloride, put some of it in a cavity in a piece of charcoal, and cover it with an equal volume of anhydrous sodium carbonate. Heat the mixture in a reducing flame of a blow pipe and when there is space in the cavity add the remainder of the silver chloride and more sodium carbonate. Finally heat the mixture until metallic globules appear. Poke these together and fuse them into a single mass. (1) What is the metal? (2) By what kind of process was it formed? (3) What was the agent? (4) Was any oxide deposited about the sides of the cavity, as in the case of lead? (5) Why?

EXPERIMENT 138. **Cupric Compounds.**

Reference: **724, 729.**

DISCUSSION: When sodium hydroxide is added to a solution of a cupric salt, cupric hydroxide is precipitated. By trying the action of this compound with an excess of sodium hydroxide, evidence is gained as to whether or not copper exhibits acid-forming properties, as do many of the metals already studied. Cupric hydroxide is rather unstable, so that even below 100° it is partially dehydrated.

As has been noted in numerous cases already, ammonium hydroxide often differs from sodium hydroxide in its action upon salts, particularly when present in excess. In the case of cupric salts the ammonia present in the solution forms a complex ion of the formula $Cu(NH_3)_4{}^{++}$.

Cupric ferrocyanide, $Cu_2Fe(CN)_6$, is sufficiently characteristic in appearance to be serviceable in testing for cupric salts.

MATERIALS: Solutions of cupric sulphate, and of potassium ferrocyanide.

DIRECTIONS: (*a*) To 5 c.c. of cupric sulphate solution add sodium hydroxide solution a few drops at a time and, finally, in excess. (1) Name and describe the precipitate. (2) Did it react with an excess of sodium hydroxide? (3) What do you conclude as to the acid-forming properties of copper? (4) Why should this be anticipated from the position of copper in the periodic classification?

Heat the contents of the test tube to boiling. (5) How did the precipitate change? (6) What is the nature of the reaction?

(*b*) To 5 c.c. of cupric sulphate solution add a few drops of ammonium hydroxide and then an excess of this reagent. (1) What was the precipitate first formed? (2) What was the final appearance of the solution? (3) To what ion is the color due? (4) Write equations for the reaction caused by the first addition of ammonium hydroxide and for the one with the excess of the reagent. (5) With compounds of what metals do similar reactions take place?

(*c*) To 5 c.c. of cupric sulphate solution add a few drops of potassium ferrocyanide solution. (1) What happened? (2) Write an equation for the reaction. (3) In what procedure is this reaction employed? (4) What other reaction is used for this purpose?

EXPERIMENT 139. **Cuprous Compounds.**

Reference: **723, 725, 726.**

DISCUSSION: When soluble cupric compounds are reduced in alkaline solution, cuprous oxide is precipitated. Since ordinary cupric salts can not exist in an alkaline solution, because of the precipitation of cupric hydroxide, complex compounds must be used. Such a compound is formed by adding a solution containing sodium potassium tartrate and sodium hydroxide to one of cupric sulphate. When this mixture, known as Fehling's solution, is boiled with a reducing agent, such as glucose, cuprous oxide is precipitated.

Cuprous chloride can be prepared by the reduction of cupric chloride by copper in the presence of concentrated hydrochloric acid. The soluble compound, H_2CuCl_3, at first formed under these conditions, decomposes upon dilution, with the precipitation of the simple chloride.

The oxidizing action of cupric compounds is exemplified in the action between a soluble cupric salt and potassium iodide, where cuprous iodide and iodine are formed instead of cupric iodide.

MATERIALS: Fehling's solution, solutions of glucose, of cupric chloride, of cupric sulphate, of potassium iodide, concentrated hydrochloric acid, copper filings, starch.

DIRECTIONS: (*a*) Mix 5 c.c. of Fehling's solution number 1 with 5 c.c. of Fehling's solution number 2. (1) What does each of these solutions contain? (2) What is the appearance of the copper compound produced? (3) What is the nature of its composition?

Add 1 c.c. of glucose solution and boil the mixture. (4) Name and describe the precipitate. (5) What kind of process has the copper undergone? (6) What was the agent?

(*b*) Dissolve 2 grams of cupric chloride in 15 c.c. of water in an Erlenmeyer flask, add 3 c.c. of concentrated hydrochloric acid and 5 grams of copper turnings and boil the solution in the hood until the green tint is no longer perceptible in the liquid. (1) What is the formula of the copper compound in solution? (2) What kind of process has the copper undergone? (3) What was the agent?

Pour the liquid into a large test tube nearly full of water. (4) Name and describe the precipitate. (5) Write two equations to show the steps by which it was formed from cupric chloride. (6) If the water assumes a slightly bluish color, what is the cause? Allow the sunlight to fall upon the precipitate. (7) What change in color is observable? (8) The chloride of what other metal resembles this compound in solubility and in change in sunlight? (9) Why might this similarity have been anticipated?

(*c*) Dilute 3 c.c. of cupric sulphate solution with 3 c.c. of water, add 3 c.c. of potassium iodide solution and filter. (1) Name and describe the precipitate.

Add a few drops of the filtrate to a test tube half full of starch solution. (2) What was the result? (3) What was in the filtrate? (4) By what kind of process was it formed? (5) What was the agent? (6) Write an equation for the reaction. (7) In what previous experiment was the precipitate formed by a similar process?

EXPERIMENT 140. **Preparation of Cupric-ammonia Sulphate.**

Reference: See Experiment 111.

DISCUSSION: A number of the soluble salts formed as the result of the addition of ammonia to the salts of copper crystal-

lize well and can be prepared readily. The compound formed from copper sulphate has the composition represented by the formula $Cu(NH_3)_4SO_4,H_2O$.

MATERIALS: Copper sulphate.

DIRECTIONS: Place 20 grams of finely powdered copper sulphate, $CuSO_4,5H_2O$ in a small flask and, under the hood, pour upon it 25 c.c. of concentrated ammonia. Shake the flask for about 1 minute and then heat it gently until all the solid has dissolved. Place the flask in a large beaker containing cold water. When the solution has cooled, hold the flask under running water. Filter off the crystals. Press them with the end of a rod and let them drain. Remove the filter paper, open it, and place it on two or three sheets of filter paper. Press the crystals with a piece of paper and set them aside to dry. (1) Weigh the crystals. (2) Calculate the weight of the compound that should be formed from the amount of copper sulphate used. (3) Calculate the percentage of this amount obtained in the experiment.

IRON

EXPERIMENT 141. **Tests for Iron.**

Reference: **751, 753, 756.**

DISCUSSION: Iron forms complex cyanides with potassium cyanide, both in the ferrous and ferric conditions, in which it is part of the negative complex radicals. These radicals form salts, which are highly colored, in one case with ferric iron as the positive part of the salt and in another with ferrous iron. Since most ferrous salts are easily oxidized by the air, they are seldom free from ferric compounds. Ferrous ammonium sulphate, however, is less easily oxidized than the simple ferrous sulphate and is therefore used in this experiment. Ferric thiocyanate is also highly colored and is thus serviceable as a test for ferric salts.

MATERIALS: Solutions of ferric chloride, of ferrous ammonium sulphate, of potassium ferrocyanide, of potassium ferricyanide, of ammonium thiocyanate.

DIRECTIONS: Put 5 c.c. of ferric chloride solution into a small test tube, fill the tube with water, shake the solution and then pour a third of it into each of two other test tubes. Dilute and divide into thirds in the same way 5 c.c. of ferrous ammonium sulphate solution. Add a few drops of solutions of potassium ferrocyanide, of potassium ferricyanide, and of ammonium thiocyanate to the three solutions of ferric chloride respectively and likewise to the three solutions of ferrous ammonium sulphate. (1) Tabulate the results obtained in these six tests. (2) Do the results indicate the presence of any ferric salt in the ferrous salt? Would you expect the production of any color in the absence of ferric salts? (3) State one test for a ferrous salt and two tests for a ferric salt. (4) Write equations for the reactions taking place in the six test tubes.

EXPERIMENT 142. Reduction and Oxidation of Iron Salts.

Reference: **127, 128, 752.**

DISCUSSION: Soluble ferric salts may be reduced by various reagents such as stannous chloride, by metallic iron in the presence of acids, and by nascent hydrogen. In this connection the difference in activity between hydrogen gas and nascent hydrogen can be well illustrated.

MATERIALS: Solutions of ferric chloride, of stannous chloride, of potassium ferrocyanide, of potassium ferricyanide, iron, zinc.

DIRECTIONS: (*a*) To 5 c.c. of ferric chloride solution add 5 c.c. of stannous chloride solution. Test a portion of the solution for a ferrous salt. (1) How did you test? (2) With what result? (3) Write an equation for the reaction.

(*b*) To 5 c.c. of ferric chloride solution add 5 c.c. of dilute hydrochloric acid and 5 grams of iron filings. After five minutes, test a portion of the solution for a ferrous salt. (1) What was the result of the test? (2) Write an equation for the original reaction. (3) With what chloride have you carried out a similar experiment and with what result? (4) Which chloride would you expect to be formed when iron is treated with hydrochloric acid? (5) Why?

Verify your prediction by treating 5 grams of iron filings with 10 c.c. of dilute hydrochloric acid and after five minutes testing portions of the solution for a ferrous salt and for a ferric salt. (6) What were the results of the tests? (7) Did your prediction prove correct? (8) Was there any evidence to the contrary? (9) If so, how do you account for it?

(*c*) Set up an apparatus for generating hydrogen from hydrochloric acid with zinc and bubble the hydrogen into 5 c.c. of ferric chloride solution for three minutes. Test some of the solution for a ferrous salt. (1) What was the result of the test? (2) Will hydrogen gas reduce a ferric salt in solution?

(*d*) To 5 c.c. of ferric chloride add 5 c.c. of dilute hydrochloric acid and a strip of zinc. After five minutes, test a portion of the solution for a ferrous salt. (1) What was the result of the test? (2) Had the ferric chloride been reduced? (3) What was the agent? (4) Write an equation for the

reaction. (5) How do you account for the difference between the result in this case and in part (*c*)?

(*e*) To 5 c.c. of a solution of ferrous ammonium sulphate add 5 c.c. of dilute nitric acid and heat to boiling. Test the solution for the presence of a ferrous salt. (1) What did you use and with what result? (2) What happened to the ferrous salt? (3) What was the agent?

Try to bring about this same kind of change by using some other reagent than nitric acid. (4) What did you use? (5) How did you prove that the desired reaction had been accomplished?

EXPERIMENT 143. **The Preparation of Ferric Ammonium Alum.**

Reference: **752, 688.**

DISCUSSION: The sulphates of the trivalent metals form double salts with the sulphates of ammonium and the alkali metals. All these salts have analogous compositions which are represented by the general formula:

$$\overset{\text{I}}{M}_2SO_4,\overset{\text{III}}{M}_2(SO_4)_3,24H_2O$$

They are isomorphous with one another, crystallize in octahedra, and are called alums. In the experiment described below ferric ammonium alum is prepared from ferrous sulphate and ammonium sulphate. The ferrous sulphate is first oxidized by means of nitric acid to ferric sulphate.

MATERIALS: Ferrous sulphate, ammonium sulphate, concentrated nitric acid, materials used in testing for ferric and for ammonium compounds.

DIRECTIONS: Place 28 grams of crystalline ferrous sulphate, $FeSO_4,7H_2O$ in a beaker and add 25 c.c. of water and 5 c.c. of concentrated sulphuric acid. Heat the mixture until the salt dissolves. Support the beaker on a tripod under the hood and add cautiously in small portions 10 c.c. of concentrated nitric acid. Heat the solution cautiously and boil it for 5 minutes. (1) What does the change in color of the solution indicate? (2) Write the equation for the reaction involved.

Dissolve 13 grams of ammonium sulphate in 25 c.c. of boiling water and filter if necessary. Mix the two solutions and set aside the beaker until the next exercise. (3) Describe the form of the crystals.

Filter off the crystals, spread them on a filter paper and when they are dry weigh them. (4) Record the weight. Determine if the salt contains water of crystallization and (5) state what you did to find out. Apply a test to determine if the salt contains ferric iron and (6) state what you did and your conclusion. Test the crystals for an ammonium compound. (7) What did you do and with what result? (8) Give the name and formula of the crystals. (9) To what class of substances do they belong? (10) Write the formulas for two other substances which are isomorphous with this compound.

CHROMIUM

EXPERIMENT 144. **Chromic Anhydride, Chromates, and Dichromates.**

Reference: **777.**

DISCUSSION: As was stated in Experiment 93, it is often convenient to regard a salt as composed of the oxide of a metal and of the oxide of a non-metal, that is, of an acid anhydride. Thus one molecule of chromic anhydride with one of potassium oxide may be regarded as forming the combination K_2O,CrO_3 or K_2CrO_4, potassium chromate, and two of chromic anhydride with one of potassium oxide as forming $K_2O,2CrO_3$ or $K_2Cr_2O_7$, potassium dichromate.

When potassium chromate is treated with concentrated sulphuric acid, the chromic acid, which would be formed by double decomposition, becomes dehydrated by the concentrated sulphuric acid and chromic anhydride is produced. A similar reaction takes place when potassium dichromate is treated with concentrated sulphuric acid.

MATERIALS: Solutions of potassium dichromate, of potassium chromate, of concentrated sodium hydroxide, potassium dichromate, glass wool.

DIRECTIONS: (*a*) Powder in a mortar 1 gram of potassium dichromate, transfer it to a test tube, add 5 c.c. of water and heat it to boiling. Cool the tube under the tap, allow the crystals to settle and pour off the saturated solution into a small beaker. To this add 10 c.c. of concentrated sulphuric acid and when the liquid has again become cold, filter out the precipitate through a funnel into the tip of which has been forced some glass wool. (**1**) Name and describe the precipitate. (**2**) Write an equation for its formation. (**3**) Why was paper not used for filtering?

Rest the funnel in a clean test tube and pour over the

precipitate drop by drop 2 c.c. of *concentrated* sodium hydroxide solution. (4) What color is the liquid? (5) What does this lead you to suppose the compound to be? (6) Write an equation for its formation.

(*b*) To 5 c.c. of potassium chromate solution add 5 c.c. of sulphuric acid. (1) What change in color occurred? (2) How do you account for it?

(*c*) To 5 c.c. of potassium dichromate solution add sodium hydroxide slowly until a change of color appears. (1) What was the change? (2) Write an equation for the reaction. (3) What are the relations in composition among chromic anhydride, a chromate, and a dichromate?

EXPERIMENT 145. **Reduction of Potassium Dichromate.**

Reference: **775, 779.**

DISCUSSION: Potassium dichromate can be reduced by ferrous salts in acid solution. This reaction is frequently used for the oxidation of ferrous salts in the course of quantitative analysis. In writing equations for the reduction of a dichromate, it is convenient again to regard the compound from the point of view of chromic anhydride and to write hypothetical steps in the reaction. The anhydride loses oxygen and the resulting chromic oxide and potassium oxide are converted into salts by the acid. The oxygen furnished in this way accomplishes the oxidation.

Potassium dichromate will oxidize oxalic acid, $C_2H_4O_2,2H_2O$, in sulphuric acid solution to water and carbon dioxide. Upon evaporating the resulting solution of potassium and chromic sulphates, a double compound of these salts with twenty-four molecules of water of crystallization, which belongs to the class of alums, separates out in well-defined crystals. If the solution is heated, however, a green compound of very different properties is formed and no crystals can be obtained.

MATERIALS: Solutions of ferrous ammonium sulphate, of potassium dichromate, of potassium ferrocyanide, of potassium ferricyanide; potassium dichromate, oxalic acid.

DIRECTIONS: (*a*) To 10 c.c. of ferrous ammonium sulphate solution add 10 c.c. of water, 5 drops of concentrated sulphuric

acid, and 10 c.c. of potassium dichromate solution. Test a small amount of the solution for a ferrous salt, and if any is present, add successive portions of the dichromate solution of 5 c.c. each until no ferrous salt remains. Test the solution for a ferric salt. (1) What test did you make and with what result? (2) Write equations for the reaction in three hypothetical steps and then combine them into a single equation. (3) In what analytical process is this reaction employed? (4) Explain how it is used.

(*b*) (1) Write equations in steps for the reduction of potassium dichromate by oxalic acid in the presence of sulphuric acid and combine the equations into a single one. (2) Calculate the weight of crystallized oxalic acid required to react with 5 grams of potassium dichromate and (3) the volume of sulphuric acid (Sp. gr. = 1.84) required.

Add 5 grams of potassium dichromate to 35 c.c. of hot water in a beaker, and when it has dissolved, cool the solution, and add the calculated amount of sulphuric acid. Place the beaker in cold water in a pneumatic trough and add two grams more than the calculated weight of oxalic acid, a few crystals at a time, with constant stirring and with care that the solution remains cold. (4) What color is the solution?

Pour 5 c.c. of the solution into a test tube and heat it to boiling. (5) What color is the solution? (6) Why was the solution cooled during the reaction? Transfer the remainder of the solution to an evaporating dish and set it aside in your desk until the next exercise, when crystals should have begun to form. Pick out with the pincers four of the largest crystals, put them in a small beaker, and pour over them the solution, taking care that no other crystals get into the beaker. (7) Give the name and write the formula of these crystals. Again set the solution aside to allow the crystals to grow and at each exercise remove the small crystals. In this way very large well-formed crystals may be produced.

MANGANESE

EXPERIMENT 146. **Potassium Permanganate as an Oxidizing Agent.**

Reference: **794.**

DISCUSSION: Potassium permanganate is a useful oxidizing agent in quantitative analysis. In neutral or alkaline solution the manganese is reduced from the valence of seven to the valence of four, thus furnishing oxidation to the extent of three valences, but in acid solution it is reduced to the valence of two, furnishing five valences for oxidation.

MATERIALS: Solutions of ferrous ammonium sulphate and of potassium permanganate.

DIRECTIONS: (*a*) To 5 c.c. of ferrous ammonium sulphate solution add potassium permanganate solution very slowly, a few drops at a time toward the end, until a slight excess is present, as indicated by the color of the solution. (1) What is the precipitate? (2) Write hypothetical equations, similar to those written for potassium dichromate in Experiment 145, to show the composition of potassium permanganate as being potassium oxide and an acid anhydride, and to show the reduction of the anhydride as it occurs in alkaline or neutral solution. (3) Write equations for the remainder of the hypothetical steps in the oxidation of the ferrous salt and combine them into a single equation for the reaction.

(*b*) To 5 c.c. of ferrous ammonium sulphate solution add 10 drops of concentrated sulphuric acid and then potassium permanganate solution as in part (*a*). (1) What is the appearance of the solution? (2) Write an equation, similar to the one you wrote in part (*a*), to show the amount of oxidation furnished by potassium permanganate in acid solution. (3) Complete the equations for the reaction with the ferrous sulphate.

INDIVIDUAL PROBLEMS

Individual assignments for the identification and preparation of compounds are made during the latter part of the year. Work on these problems should be done only after the *class* work for the day has been finished.

IDENTIFICATION OF COMPOUNDS

DISCUSSION: Application of the tests studied in many of the preceding experiments and of tests now to be used for the first time is made by applying these tests to compounds given out individually for identification. Single compounds only will be issued, since the analysis of mixtures often requires a process of separation, involving a systematic scheme of qualitative analysis, before tests can be made.

The procedure in general, after noting the appearance of the substance, is to get the material into solution, first trying water alone and subsequently, if necessary, acids. The metallic element of the compound is identified first. An indication of what this may be is obtained by adding ammonium hydroxide to the solution, and then a confirmatory test is tried to prove definitely the presence of the element suspected. From the knowledge of the solubility of the compound and of the metallic element which it contains, a conclusion may be drawn by reference to the table of solubilities, as to what acid radical may be present. The addition of silver nitrate and of barium chloride to portions of the solution causes the precipitation of certain groups of acid radicals if present, and thus offers additional basis for judging what radicals must be tested for. Finally a few confirmatory tests will identify the acid radical and consequently the compound.

Often the acid radical can be recognized when the compound is first dissolved, particularly when a gas is evolved in the process.

Many opportunities for thoughtless misinterpretation exist in making these tests, as for example in testing for a chloride when the compound has been dissolved in hydrochloric acid. A student can frequently save himself needless work by thoughtful interpretation of his results.

Complete notes must be kept, since a negative result in making a test is as important as a positive one. Abbreviations and symbols may be used, as illustrated in the following sample report, which is given as a suggestion for the form of notes.

No. 6 April 6, 1922.

White powder, insol. water, sol. cold dil. HNO_3 with effervescence.

Metal.

NH_4OH = wh. ppt. insol. excess, = Pb, Hg, Bi, Sb, Sn, Al or Mg.

HCl and H_2S = black ppt., = Pb, Hg, or Bi.

HCl = . . . H_2SO_4 = . . . Not Pb.

$SnCl_2$ = ... Not Hg.

NH_4OH ppt. dissolved in 2 drops conc. HCl, water gave wh. ppt. = Bi

Acid Radical.

Table of sol. HF, H_2SO_4, HCl, HBr, $HClO_3$, HNO_3 impossible.

HCl, to solid salt, gas, colorless, odorless.

$Ca(OH)_2$ on rod milky = CO_2.

Report, bismuth carbonate.

DIRECTIONS: Procure at the storeroom the substance assigned for identification by giving your name and the number of the salt you want, e.g., first, second, etc. A little of the salt should always be reserved until after the report on the analysis has been made, so that the instructor may test the *original* sample, if a test is necessary. A second sample of any salt may be got if the first has been used up before a satisfactory result has been obtained.

Examine and record the appearance of the substance. If it is not already powdered, grind it in the mortar. Try a pinch of the substance with each of the following solvents until one is found which will dissolve it: cold water; boiling water; cold, dilute hydrochloric acid; boiling, dilute nitric acid; hot aqua regia (three parts of concentrated hydrochloric acid and one part of concentrated nitric acid). When using concentrated acids avoid large quantities. Sulphides which dissolve in boiling, dilute nitric acid will leave a ball of spongy sulphur on the surface, which, in itself, serves as a test for a sulphide. When a suitable solvent has been found, get a considerable quantity of the substance into solution, using about one spatulaful to 50 c.c. of solvent.

If acid has been used to dissolve the salt, neutralize as nearly as possible with *sodium* hydroxide, since the presence of ammonium salts prevents the precipitation of certain metals by ammonium hydroxide. Do not allow a permanent precipitate to form, even if the solution has to be left slightly acid. If a precipitate forms, redissolve it with a few drops of the acid.

IDENTIFICATION OF THE METAL

To 5 c.c. of the solution add ammonium hydroxide drop by drop and finally in excess. Record the result and the conclusion to be drawn in accordance with the facts given below as to the action with ammonium hydroxide.

If no precipitate appears, try the flame test by moistening the platinum wire in hydrochloric acid, dipping it into the salt to be tested and then into the flame. This will identify barium and potassium. Calcium and strontium, however, give flames so nearly alike that the confirmatory tests must always be applied. A yellow flame does not indicate sodium unless it persists ten or fifteen seconds, as sodium compounds are present as impurities in nearly all chemicals. Never report sodium unless the test for potassium has first been applied. Absence of flame test indicates arsenic or ammonium compounds.

If ammonium hydroxide gives a white precipitate insoluble in excess, acidify a fresh portion of the solution with hydrochloric acid and pass in hydrogen sulphide. A black precipitate indicates lead, mercuric mercury, or bismuth, which should be tested for only in the order given. An orange precipitate indicates antimony; a yellow or brown one, tin; and no precipitate, aluminium or magnesium. A dirty white precipitate of sulphur sometimes is formed, which has no significance.

ACTION WITH AMMONIUM HYDROXIDE

No Precipitate. Arsenic, barium, strontium, calcium, sodium, potassium, or ammonium.

White Precipitate, soluble in excess. Cadmium or zinc.

White Precipitate, insoluble in excess. Lead, mercury (mercuric), bismuth, antimony, tin, aluminium, magnesium.

Blue-Green Precipitate. Chromium.

Blue Precipitate, soluble in excess to a brown solution. Cobalt.

Blue Precipitate, soluble in excess to a dark blue solution. Copper.

Light, Dirty Green Precipitate. Iron (ferrous).

Green Precipitate, soluble in excess to a light blue solution. Nickel.

Brownish White Precipitate, turning darker on exposure. Manganese.

Red-Brown Precipitate. Iron (ferric).

Brown Precipitate, soluble in excess to a colorless solution. Silver.

Black Precipitate. Mercury (mercurous). Some iron salts also give a black precipitate.

CONFIRMATORY TESTS FOR METALS

Make confirmatory tests for the element or elements indicated as probably present.

Aluminium. Ammonium chloride and ammonium hydroxide give a white precipitate of aluminium hydroxide, insoluble in excess; soluble in sodium hydroxide.

Ammonium. Heated with sodium hydroxide, ammonia is formed, which can be detected by the odor or by moist litmus paper held in the gas.

Antimony. Hydrogen sulphide precipitates orange antimony sulphide in the presence of dilute hydrochloric acid. The sulphide is soluble in yellow ammonium sulphide, and soluble in hot, concentrated hydrochloric acid.

Arsenic. Hydrogen sulphide precipitates yellow arsenic sulphide in the presence of dilute hydrochloric acid. The sulphide is soluble in yellow ammonium sulphide, insoluble in hot, concentrated hydrochloric acid.

Barium. Imparts green color to the flame. Sulphuric acid precipitates white barium sulphate.

Bismuth. Hydrogen sulphide precipitates black sulphide in presence of dilute hydrochloric acid.

Ammonium hydroxide precipitates white hydroxide which, when filtered, washed, and dissolved on the filter with two or three drops of concentrated hydrochloric acid, and poured into water, gives a white precipitate of bismuth oxychloride.

Cadmium. Hydrogen sulphide precipitates yellow cadmium sulphide in presence of dilute hydrochloric acid, which is insoluble in yellow ammonium sulphide.

Calcium. Gives a dull red color to the flame. Gives no precipitate with calcium sulphate (distinction from strontium).

Chromium. Mixed with sodium carbonate and potassium nitrate and fused in a platinum wire loop gives yellow potassium chromate. A solution of the bead in acetic acid, gives yellow precipitate with lead acetate.

Cobalt. Borax bead colored deep blue.

Copper. Hydrogen sulphide precipitates black copper sulphide in presence of dilute hydrochloric acid. Salts give green flame. Potassium ferrocyanide gives brown precipitate of copper ferrocyanide in presence of acetic acid.

Iron. Potassium ferricyanide gives blue color with ferrous salts. Potassium ferrocyanide gives blue color with ferric salts and potassium thiocyanate gives red color with ferric salts.

Lead. Hydrochloric acid precipitates from solutions not too dilute, white lead chloride, soluble in hot water.

Sulphuric acid precipitates white lead sulphate.

Magnesium. No precipitate with ammonium chloride and ammonium hydroxide. Disodium phosphate added to the mixture precipitates white, crystalline magnesium ammonium phosphate.

Manganese. Ammonium sulphide precipitates flesh-colored manganese sulphide.

Compounds when fused with sodium carbonate and potassium nitrate in platinum wire loop, give green potassium manganate.

Mercury. *Mercuric.* Stannous chloride precipitates white mercurous chloride or gray mercury.

Mercurous. Hydrochloric acid precipitates white mercurous chloride, which is converted into a black compound by ammonium hydroxide.

Nickel. Salts are usually green. Compounds color borax beads violet when hot, red-brown when cold.

Potassium. Violet flame. If flame is yellow, look through two pieces of cobalt glass to detect the reddish color in the flame. Do not be misled by the color of the heated wire.

Silver. Hydrochloric acid precipitates white silver chloride, soluble in ammonium hydroxide.

Sodium. Yellow flame persisting for ten or twenty seconds. Before reporting sodium, prove the absence of potassium and ammonium by proper tests, since an impurity of sodium gives a strong flame.

Strontium. Bright red flame. White precipitate with calcium sulphate, forming slowly in concentrated solution. (Compare with calcium.)

Tin. *Stannic.* Hydrogen sulphide precipitates yellow stannic sulphide in presence of dilute hydrochloric acid. Precipitate soluble in yellow ammonium sulphide, soluble in hot, concentrated hydrochloric acid (distinction from arsenic).

Stannous. Hydrogen sulphide precipitates brown stannous sulphide in the presence of dilute hydrochloric acid. Precipitate soluble in yellow ammonium sulphide.

Zinc. In acetic acid solution, i.e., containing no strong acid or alkali, hydrogen sulphide precipitates white zinc sulphide.

IDENTIFICATION OF THE ACID-RADICAL

When the metallic element has been identified, determine by reference to the table of solubilities on page 543 what acid radicals may be present in accordance with the solubility and color of the compound.

To a small portion of the solid substance add a few cubic centimeters of dilute hydrochloric acid and heat. If a gas is evolved, identify it by its color, its odor, its action with lime water, or with lead acetate paper. If the compound is insoluble in the acid and if the previous results indicate the possibility of a sulphide, add a strip of zinc to the hydrochloric acid mixture and test for hydrogen sulphide.

If no gas is evolved, the presence or absence of a halide may be ascertained by adding silver nitrate to a nitric acid solution of the compound.

If ammonium hydroxide gave no permanent precipitate with a solution of the compound, barium chloride may be added to such an alkaline solution and the presence or absence of a sulphate, phosphate, borate, or fluoride is indicated by whether or not a precipitate appears. When, however, the metallic element of the compound is one which forms an insoluble chloride, barium nitrate must be substituted for barium chloride.

The procedure described above will have identified the acid radical present or have eliminated a large number of possibilities. In the former case a confirmatory test with a fresh portion of the compound should be made, and in the latter case confirmatory tests for all the remaining possibilities should be tried.

TESTS FOR THE ACID-RADICALS

Acetate. (*a*) The salt heated with a few drops of concentrated sulphuric acid gives the characteristic odor of acetic acid.

(*b*) A pinch of the salt heated with five drops of concentrated sulphuric acid and 1 c.c. of alcohol gives a fruity odor, due to the formation of ethyl acetate.

Borate. Turmeric paper, after having been dipped in a hydrochloric acid solution of the salt, turns bright pink upon drying at 100° and dark green upon subsequent treatment with ammonium hydroxide.

Bromide. One c.c. of carbon disulphide added to 5 c.c. of the solution is turned cherry red after 3 c.c. of chlorine water has been added and the mixture shaken.

Carbonate. Dilute hydrochloric acid causes the evolution of an odorless gas, which gives a precipitate with lime water.

Chlorate. Silver nitrate gives no precipitate in a nitric acid solution; but a pinch of the salt heated strongly in a dry test tube, cooled and dissolved gives a precipitate with silver nitrate.

Chloride. Silver nitrate gives in nitric acid solution a white precipitate, which dissolves readily in ammonium hydroxide.

Fluoride. To a pinch of the salt in a dry test tube is added an equal amount of sand and the mixture is moistened with concentrated sulphuric acid and gently heated. A platinum wire with a drop of water adhering to a loop in the end, is carefully thrust into the tube above the mixture. A white precipitate in the drop of water shows the presence of a fluoride.

Hydroxide or Oxide. All acid radicals having been proved absent, the compound may be reported as an oxide or hydroxide. The latter may sometimes be distinguished by heating a little of the compound in a dry test tube, when a small deposit of moisture will collect in the upper part of the tube.

Iodide. A deep blue color is produced upon adding to the solution a few drops of starch solution and a few drops of chlorine water.

Nitrate. When to 5 c.c. of solution is added 5 c.c. of ferrous sulphate solution and when 5 c.c. of concentrated sulphuric acid is carefully poured down the side of the test tube, a brown ring appears. This test is of value only in the absence of a nitrite.

Nitrite. Dilute sulphuric acid evolves a brown gas from the solid when it is heated.

Phosphate. Three c.c. of ammonium molybdate, heated with 1 c.c. of the solution to which has been added 2 c.c. of dilute nitric acid, produces a yellow precipitate.

Silicate. A few drops of concentrated hydrochloric acid produce a gelatinous precipitate when added to a few cubic centimeters of a concentrated solution of the salt.

Sulphate. Barium chloride gives a white precipitate, insoluble in hydrochloric acid.

Sulphide. Hydrochloric acid, or hydrochloric acid and zinc, evolves hydrogen sulphide, recognizable by its odor and by its action on lead acetate paper.

Sulphite. Hydrochloric acid evolves sulphur dioxide, recognizable by its odor.

Thiosulphate. Hydrochloric acid evolves sulphur dioxide and at the same time produces a precipitate of sulphur.

APPENDIX

VAPOR PRESSURES OF WATER

The temperatures are given in degrees centigrade, and the pressures in millimeters of mercury.

Temp.	Press.	Temp.	Press.	Temp.	Press.	Temp.	Press.
0°	5	14°	12	20°	17	26°	25
4	7	15	13	21	19	27	27
8	8	16	13	22	20	28	28
10	9	17	14	23	21	29	30
12	10	18	15	24	22	30	32
13	11	19	16	25	24	100	760

ELECTROMOTIVE SERIES OF THE ELEMENTS

Potassium
Sodium
Barium
Strontium
Calcium
Magnesium
Aluminium
Manganese
Zinc
Chromium
Cadmium
Iron
Cobalt
Nickel
Tin
Lead
Hydrogen
Copper
Arsenic
Bismuth
Antimony
Mercury
Silver
Palladium
Platinum
Gold

. . . . . .

Iodine
Bromine
Oxygen (O^{--})
Chlorine
Oxygen (OH^{-})

TABLE OF SOLUBILITIES

	Acetate	Borate	Bromide	Carbonate	Chlorate	Chloride	Fluoride	Hydroxide	Iodide	Nitrate	Nitrite	Phosphate	Silicate	Sulphate	Sulphide	Sulphite	Thiosulphate
*Aluminium	W	A	W	..	W	W	A	A	W	W	..	A	AI	W	A	A	..
Ammonium	W	W	W	W	W	W	W	W	W	W	W	W	..	W	W	W	W
*Antimony	..	..	WA	..	..	WA	W	A	WA	..	..	WA	..	WA	A	..	..
Arsenic	..	..	W	..	..	W	W	A	W	..	..	A	..	..	A	..	..
Barium	W	A	W	A	W	W	WA	W	W	W	W	A	A	I	W	A	WA
*Bismuth	W	A	WA	A	W	WA	A	A	A	W	..	A	..	W	A	A	..
Cadmium	W	WA	W	A	W	W	WA	A	W	W	W	A	A	W	A	WA	W
Calcium	W	WA	W	A	W	W	AI	WA	W	W	W	A	A	WI	WA	A	W
*Chromium	W	A	WI	..	W	WI	W	A	W	W	..	A	A	W	AI	A	..
Cobalt	W	A	W	A	W	W	WA	A	W	W	..	A	A	W	A	A	W
*Copper	W	A	W	A	W	W	A	A	W	W	..	A	A	W	A	WA	..
Iron++	W	A	W	A	W	W	WA	A	W	W	..	A	A	W	A	A	W
*Iron+++	W	A	W	..	W	W	W	A	W	W	..	A	A	W	A	A	..
Lead	W	A	WI	A	W	WI	A	A	WA	W	W	A	A	AI	A	A	A
Magnesium	W	A	W	A	W	W	AI	A	W	W	W	A	A	W	A	W	W
Manganese	W	A	W	A	W	W	A	A	W	W	..	A	A	W	A	A	W
*Mercury+	W	..	AI	A	W	AI	..	..	A	W	..	A	..	WA	A	A	..
*Mercury++	W	..	W	A	W	W	WA	..	A	W	W	A	..	W	A	A	..
Nickel	W	A	W	A	W	W	WA	A	W	W	W	A	A	W	A	A	W
Potassium	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W
Silver	W	A	I	A	W	I	W	..	I	W	WA	A	..	WA	A	A	W
Sodium	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W
Strontium	W	A	W	A	W	W	WA	W	W	W	W	A	A	I	W	A	W
Tin++	W	A	..	..	W	W	W	A	W	..	..	A	..	W	A	A	..
*Tin++++	W	..	..	..	..	W	W	AI	W	..	..	A	..	..	A	..	..
Zinc	W	A	W	A	W	W	WA	A	W	W	W	A	A	W	A	W	W

W = sol. in water.
WA = slightly sol. in water; sol. in acids.
A = insol. in water; sol. in acids.
WI = slightly sol. in water and in acids.
I = insol. in water and in acids.
. . = unknown.
AI = insol. in water; slightly sol. in acids

* Salts (especially of weak acids) hydrolyze in water, forming precipitates unless excess of free acid is present.

APPARATUS

LABORATORY EQUIPMENT

Balance, beam
Balance, platform
Barometer
Clamps
Copper wire
Corks
Cork borers
Cork press
Crock for waste
Funnel support
Generator, Kipp
Litmus paper
Rings
Ringstand
Splinters
Spoon, deflagrating
Thread
Trough, pneumatic
Voltmeter

INDIVIDUAL EQUIPMENT

5 Beakers, Nos. 1–5
1 Blowpipe
1 Blowpipe tube
4 Bottles, gas, 250 c.c.
1 Bunsen burner
1 Cloth
1 Crucible and cover
1 Cylinder, graduated
1 Evaporating dish, 8 cm.
4 Flasks
  1 Erlenmeyer, 300 c.c.
  1 Florence, 100 c.c.
  1 Florence, 500 c.c.
  1 Florence, 750 c.c.
1 Flame spreader
1 File, triangular
Filter paper, 1 pkg., 10 cm.
2 Funnels, 6 cm.
2 Glass plates, white, 5 cm.
2 Glass plates, blue, 5 cm.
Glass rod, 2½ ft.
Glass tubing, 5 ft.
3 Ignition tubes
1 Key
Matches
1 Mortar and pestle, 8 cm.
Pincers, 1 pair
1 Pinchcock
1 Platinum wire, 5 cm.
Rubber tubing, 2 ft.
2 Rubber connectors
3 Rubber stoppers, No. 5
  2, 2-hole
  1, 1-hole
1 Ruler
1 Spoon, horn
1 Sponge
18 Test tubes
  2, 6-in.
  6, 8-in.
1 Test tube brush
1 Test tube holder
1 Test tube rack
1 Thermometer, −10° to 110°
1 Thistle tube
Tongs, 1 pair
1 Triangle, clay
3 Watch glasses
  2, 4 cm.
  1, 7 cm.
Weights, 1 box
1 Wire gauze

SPECIAL EQUIPMENT

Beaker, 1000 c.c.
Bottle, 150 c.c.
Bottle, 2000 c.c.
Burette, 50 c.c.
Burner, Meker
Cylinder, graduated, 10 c.c.
Dish, lead
Electrodes, carbon
Flask, 250 c.c.
Funnel, 10 cm.
Glass wool
Pinchcock
Pipette, graduated, 10 c.c.
Retort, 250 c.c.
Sand bath
Thermometer, 360°
Weight, brass, 200 gr.

LIST OF CHEMICALS

SOLIDS

Aluminium, sheet
Ammonium chloride
Ammonium nitrate
Ammonium nitrite
Ammonium sulphate
Antimony trichloride
Arsenious oxide
Asbestos
Barium chloride
Barium hydroxide
Barium nitrate
Barium oxide
Barium peroxide
Bismuth chloride
Bleaching powder
Calcium carbonate, marble
Calcium chloride
Calcium cyanamide
Calcium fluoride
Calcium hydroxide
Calcium phosphate, primary
Calcium sulphate
Charcoal, boneblack
Charcoal, lumps
Charcoal. powdered
Copper, gauze
Copper, sheet
Copper, turnings
Copper nitrate
Copper sulphate
Copper sulphide
Ferrous sulphate
Ferrous sulphide
Graphite, powder
Iodine
Iron, nails
Iron, powder
Iron, steel wool
Lead, sheet
Lead acetate
Lead dioxide
Lead monoxide
Lead nitrate
Lead oxide, red
Magnesium, powder
Magnesium, ribbon
Magnesium carbonate
Manganese carbonate
Manganese dioxide
Mercuric oxide
Oxalic acid
Paraffin
Phosphorus, red
Phosphorus pentachloride
Potassium aluminium sulphate
Potassium bromide
Potassium chlorate
Potassium chloride
Potassium chromium sulphate
Potassium dichromate
Potassium ferrocyanide
Potassium hydrogen tartrate

Potassium hydroxide
Potassium iodide
Potassium nitrate
Potassium permanganate
Silica, sand
Silver, tarnished
Silver chloride
Sodium
Sodium aluminium sulphate
Sodium ammonium phosphate
Sodium carbonate, crystallized
Sodium chloride
Sodium chloride, pure
Sodium chloride, rock salt
Sodium hydrogen carbonate
Sodium hydrogen sulphate
Sodium hydroxide
Sodium nitrate
Sodium peroxide
Sodium sulphate, crystallized
Sodium sulphite
Sodium tetraborate, borax
Sodium thiosulphate
Starch
Strontium chloride
Strontium nitrate
Sugar
Sulphur, powder
Tin, granulated
Tin, sheet
Zinc, dust
Zinc, pure
Zinc, sheet
Zinc chloride

LIQUIDS AND SOLUTIONS

The numbers indicate the weights in grams or the volumes in cubic centimeters per liter of solution of the respective substances as commonly obtained, often in the hydrated form.

Acid, acetic, glacial
Acid, acetic (dilute), 1N, 60
Acid hydrochloric (conc.), sp. gr. 1.20, 13N
Acid, hydrochloric, 20 per cent, 469 c.c. of HCl, sp. gr. 1.20
Acid, hydrochloric, 10 per cent, 224 c.c. of HCl, sp. gr. 1.20
Acid, hydrochloric, 1N, 77.6 c.c. of HCl, sp. gr. 1.20
Acid, hydrochloric, ½N, 38.8 c.c. of HCl, sp. gr. 1.20
Acid nitric (conc.), 16N, sp. gr. 1.42
Acid, sulphuric, 1N, 27.8 c.c. of H_2SO_4, sp. gr. 1.84
Alcohol, ethyl, 95 per cent
Alcohol, methyl
Ammonium carbonate, 1N, 67+150 c.c. NH_4OH
Ammonium chloride, 1N, 53.5
Ammonium molybdate, 75+180 c.c. HNO_3
Ammonium oxalate, ½N, 35
Ammonium sulphide (dilute ammonia sp. gr. 0.90 with equiv. vol. of water, sat. with H_2S)
Ammonium sulphide, yellow $(NH_4)_2S+S$
Ammonium thiocyanate, ¼N, 19
Arsenious chloride, ¼N, 8 g. As_2O_3+10 g. NaOH, acidify with HCl
Barium chloride, ½N, 61
Benzene
Bromine

Bromine water, sat.
Calcium chloride, 1N, 56
Calcium hydroxide, 0.04N, sat.
Calcium sulphate, sat.
Carbon disulphide
Carbon tetrachloride
Chlorine water, sat.
Chloroform
Cobalt nitrate, ¼N, 36
Copper sulphate, ½N, 62
Ether
Fehling's solution No. 1, $CuSO_4,5H_2O$, 69
Fehling's solution No. 2, $KNaC_4H_4O_6,4H_2O$, 246+NaOH, 100
Ferric chloride, ½N, 27
Ferrous sulphate, ¼N, 35
Ferrous ammonium sulphate, ¼N, 98
Glucose, 50
Hydrogen peroxide, 3 per cent
Hydrogen sulphide, sat.
Lead acetate, ¼N, 47
Lead nitrate, ¼N, 41
Magnesium chloride, ½N, 51
Mercuric chloride, ¼N, 34
Mercurous nitrate, ¼N, 75+4 c.c. HNO_3+Hg
Mercury
Methyl orange, 0.5
Nickel nitrate, ¼N, 36
Phenolphthalein (1 g. in 500 c.c. alcohol)
Phosphorus trichloride
Potassium bromide, ⅕N, 24
Potassium chloride, ½N, 38
Potassium chromate, ½N, 49
Potassium dichromate, ½N, 77
Potassium dichromate, ¼N, 38
Potassium ferricyanide, ¼N, 27
Potassium ferrocyanide, ¼N, 26
Potassium hydroxide, 1N, 56
Potassium hydroxide, ½N, 28
Potassium iodide, ⅕N, 33
Potassium nitrate, ½N, 50
Potassium permanganate, 1N, 32
Potassium permanganate, 1 gr.+200 c.c. H_2SO_4
Potassium thiocyanate, ¼N, 29
Pyrogallic acid, 10 per cent, 111
Silver nitrate, ⅕N, 34
Soap, 30

Sodium carbonate (anhyd.), ½N, 26
Sodium chloride, 5.5N, sat.
Sodium chloride, ½N, 29
Sodium chromate, ¼N, 43
Sodium hydrogen sulphite, sp. gr. 1.26
Sodium hydroxide, conc., 50 per cent, 765
Sodium hydroxide, 1N, 40
Silver sulphate, ⅛N, 31
Sodium hydroxide, 10 per cent, 112
Sodum hydroxide, ½N, 20
Sodium nitrate, ½N, 43
Sodium nitrite, ½N, 37
Sodium phosphate, secondary, ½N, 55
Sodium silicate, sp. gr. 1.38
Sodium thiosulphate, ¼N, 31
Stannic chloride, 50 c.c.+500 c.c. HCl
Stannous chloride, 1N, 112+200 c.c. HCl+Sn
Strontium chloride, ¼N, 33
Sulphurous acid, sat.
Zinc chloride, ¼N, 17
Zinc sulphate, ¼N, 36

DESK REAGENTS

Ammonium hydroxide (dilute), 5N, 685 c.c. of NH_4OH sp. gr. 0.90
Hydrochloric acid (dilute), 5N, 388 c.c. of HCl, sp. gr. 1.20
Nitric acid (dilute), 5N, 317 c.c. of HNO_3, sp. gr. 1.42
Sulphuric acid (conc.), sp. gr. 1.84, 36N
Sulphuric acid (dilute), 5N, 139 c.c. of H_2SO_4, sp. gr. 1.84
Sodium hydroxide, 3N, 120